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# THE DEVELOPMENT OF HIGH ENERGY DENSITY PRIMARY BATTERIES, 200 WATT HOURS PER POUND OF TOTAL BATTERY WEIGHT MINIMUM

by

William E. Elliott, Shih-liang Hsu, and Warren L. Towle

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-2790

FINAL REPORT

August 1, 1963 - July 31, 1964

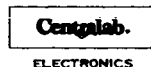
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January, 1965

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Technical Management  
Space Power Systems Division  
National Aeronautics and Space Administration  
Lewis Research Center, Cleveland, Ohio  
Mr. Robert B. King

GLOBE-UNION INC.  
Applied Research  
900 East Keefe Avenue  
Milwaukee, Wisconsin 53201

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## SUMMARY

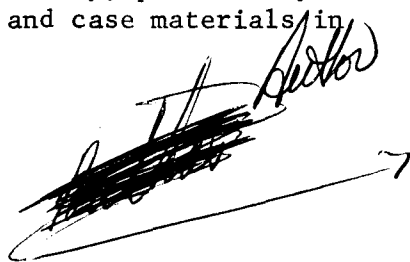
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The purpose of this contract is to develop a primary battery to deliver a minimum of 200 watt-hours per pound of battery weight. Allowing for the added weight of electrolyte, grids, case, etc., electrode couples of theoretical energy density of 500 watt-hours per pound will be needed. Approximately thirty such couples are suggested.

Aprotic electrolytes are needed to avoid reaction with the active anode materials in such systems. A program was undertaken to systematize and expand the available information on factors governing the behavior of conductive solutions. Progress has been made on elaborating the effects of such solvent properties as dielectric constant, viscosity, and the ability to solvate the ions of the solute. In addition, much information has been gained on the relative behavior of a large number of solute materials, indicating the effects of anion and cation size on solubility, conductivity, and solution viscosity.

Six aprotic solvents and over 70 solutes have been investigated. From these, over 30 solvent-solute systems with conductivity at one-molal concentration in excess of  $10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$  are now known.

The bulk of the work in this contract period has been on electrolyte systems. Also, some exploratory tests have been conducted on the behavior of cell couples in the electrolytes studied. In addition, the stability of several of the most promising electrode materials in solvents and electrolytes has been determined. Finally, preliminary stability tests have been conducted on separator and case materials in the more promising solvents.

A handwritten signature, possibly "J. H. ...", is written over a large, dark, horizontal scribble. A long, thin line extends from the bottom right of the scribble towards the right margin.

## INTRODUCTION

The purpose of this investigation is to develop a primary battery with an energy density of at least 200 watt-hours per pound of total battery weight. These batteries will operate at near-normal temperatures and pressures.

Many electrochemical systems can be postulated which should deliver sufficient energy per pound to meet this requirement, even allowing for the unavoidable "dead weight" of such items as electrolyte, grids, separators, and case. However, in order for the energy of the active electrode materials to be extracted from the battery, an electrolyte must be present in order to provide the means for electric charge and material transport to and from the electrodes. The properties of this electrolyte will exert a major role in determining energy losses due to polarization and resistance to current flow. Thus the availability of a suitable electrolyte becomes the keystone upon which the structure of the high energy battery system depends.

At the start of this contract, information on the factors governing the behavior of conductive solutions -- particularly nonaqueous solutions -- was scattered and incomplete. Furthermore, very few examples were known of electrolytes with enough conductivity to permit their use even in batteries limited to service at very low drain rates. Therefore, in order to provide a basis to guide ourselves and others in selection of materials for nonaqueous electrolytes, we undertook to clarify the picture in this field as the first step in the overall program.

THE DEVELOPMENT OF HIGH ENERGY DENSITY PRIMARY BATTERIES,  
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ABSTRACT

Electrode materials with a theoretical energy density of 500 WH/Lb are needed to enable delivery of 200 WH/Lb in a practical battery. Aprotic electrolytes must be used to avoid corrosion of the highly active anode material. We have thus far developed over thirty aprotic electrolytes with conductances in excess of  $1 \times 10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$ . We have achieved appreciable clarification of the effects of solvent properties (dielectric constant, viscosity and ability to solvate the solute ions) and solute properties on the conductance. Some preliminary tests have been conducted to study the stabilities of electrode and separator materials in aprotic solvents.

*W. L. Towle*

I. OVERALL PROGRESS

## I. OVERALL PROGRESS

### A. General Theoretical Considerations.

The development of high energy density (200 watt-hours per pound) primary batteries requires the re-evaluation of potentially usable electrode couples since commercial primary battery electrode couples do not possess sufficiently high theoretical free energy densities to meet the objective of this program. The inability to realize 100% of the energy from the active material and the presence of such "dead weight" items as separators, case, electrolyte, etc. usually result in an energy yield efficiency of no more than 40% of the theoretical energy density of the electrode couple. Thus a minimum value of 500 watt-hours per pound theoretical will be required to achieve 200 watt-hours per pound for a real system.

Some of the theoretical properties of a number of electrode couples are tabulated in Table I (page III-1) and graphically presented in Figure 1 (page III-5). See Appendix I (page IV-1) for derivation.

#### 1. Electrolyte.

Inspection of Table I (page III-1) shows that the requirement of theoretical 500 watt-hours per pound immediately limits the suitable anode materials to highly active metals. The use of such active metals precludes the use of aqueous electrolytes because reaction with water would result in wasteful corrosion of the metal and hydrogen liberation from the water. Similarly, any nonaqueous solvent containing a labile hydrogen may be attacked. Thus the use of an aprotic solvent in the electrolyte will be mandatory.

Certain other basic requirements must be met in the characteristics of an electrolyte in order to satisfy the requirements for good battery performance. The importance of the first of these, namely, the conductance, is illustrated in Figure 2 (page III-6), which shows the voltage drop resulting from internal IR loss as a function of conductance and current drain. A second factor will be the effect on the electrochemical characteristics of the electrodes themselves. Both concentration polarization and activation polarization may be influenced by the

particular electrolyte chosen. Thus for any given electrode couple it may be necessary not only to find an electrolyte of high conductance, but indeed it may be necessary to test a large number of such electrolytes in order to find one whose performance is acceptable. Therefore, in order to facilitate the task of developing new electrolyte (solvent-solute) systems, we have felt it necessary to direct our investigation toward a clarification of the basic factors governing the behavior of conductive solutions. From such a program should emerge not only a wealth of data on a wide variety of solvent-solute systems but, in addition, a clarification of the factors governing their behavior with the resultant simplification of the task of searching for additional systems.

By analogy to aqueous systems, a nonaqueous electrolyte may also be considered as a solute-solvent system. The function of the solute is to provide ions which will permit charge and material transport to and from the electrodes. The functions of the solvent are to dissolve the solute and to promote ionic dissociation thereof. Thus, in addition to providing the fluid medium for suspension of the dissociated ions, the solvent may provide one or more of the following;

- a. By virtue of a high dielectric constant it may assist in promoting the dissociation of the solute.
- b. By virtue of its coordination properties, it may provide a ligand for either the anions or cations, and thus by reduction in charge density thereof may further assist in their dissociation.

A solvent of low viscosity should be expected to favor high solution conductance by promoting ion mobility. In addition, the solvent should preferably be of low density and a liquid over the temperature range to be encountered in use. By quantitative investigation of the above factors a clarification of their effects on solute solubility and solution conductance should result.

A paper, "Investigations of High Energy Density Electrochemical Systems, Part 1 - Electrolytes," was presented at the Power Sources Conference, May, 1964. A copy of this paper is included herewith as Appendix II (page IV-3) and gives comparisons between aqueous and nonaqueous electrolytes.

## 2. Electrochemistry.

Referring to Figure 1 (page III-5) where the cell potential is plotted against the equivalent weight of electrode couples with energy density as the parameter, it is apparent that any electrochemical polarization would tend to shift the system from where it is placed downward vertically toward a lower energy density region, whereas the horizontal shift to the right depends on the efficiency of electrode material, electrolyte interactions, and the design of the battery. Since the activation polarization of the electrochemical system is a characteristic which cannot be changed by battery design, systems with low activation polarization would be desirable.

It is also essential that the products of the electrochemical reactions be either soluble or porous to permit electrolyte penetration to the active electrode material for further electrochemical reaction.

## 3. Material Stability.

- a. Electrode Stability. Both the anode and the cathode active materials must be insoluble in the solute-solvent system to prevent internal short circuiting and consequent loss in available energy.
- b. Separator Stability. The materials considered to be used as either separator material or case material of a battery must be stable in the electrolyte.

## B. Experimental Approach.

Our effort has been primarily directed toward the development of high conductance aprotic electrolytes with some supplemental qualitative electrochemical tests and material stability tests.

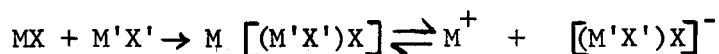
### 1. Electrolyte.

- a. Qualitative Conductance Screening Tests. This test was designed to screen out solute-solvent systems with low specific conductances from consideration as battery electrolytes and also to furnish information on the principles governing the electrolyte conductance at fairly high solute concentrations.



- b. Quantitative Determination of Conductance-Concentration Relationships. The relationships between the conductance of the electrolyte and the concentration of the solute were determined for systems which showed promising conductance in test "a". The purpose was to determine the maximum obtainable conductances for these systems.
- c. Exploratory Tests. In addition to the evaluation of the relative importance of various factors on the conductances of the electrolytes, we have also tried other lines of approach to achieve high conductance.

- (1) Salt-ether complexes. It is known that ether-oxygen is an electron donor. Since Lewis acids are electron acceptors, they should be capable of forming complexes with the ethers. The stability of the solvated metal ion portion of the resulting complex should be the highest if the metal chelate is formed in such a way that the coordination number of the metal equals the number of the ligand groups (ether-oxygen) in the ether. Very high conductance might then be obtained upon dissolving this complex in a suitable solvent. For this reason we have selected a group of ethers (Table II, page III-7) to be used in the conductance tests.
- (2) Mixed solutes. Two salts may react and form ionizable complexes which can dissociate in the solution and form free ions according to the following reactions:



If the products are easier to ionize in a solvent than either MX or M'X', an improvement in conductance should be observed.

- (3) Mixed solvents. Although low viscosity, high dielectric constant and high coordination power are the desirable properties for a good solvent, it is very unlikely that many such solvents will be found in practice. An alternative way is to use mixed solvents consisting of two solvents possessing different desirable properties. Upon varying the ratio of these two solvents, a mixed solvent with optimum properties may be obtained.

## 2. Electrochemistry.

Exploratory electrochemical tests were performed to obtain qualitative information on the magnitude and stability of the open circuit potentials of some high energy density electrode couples in order to screen out systems with poor characteristics. The quantitative determination of activation polarization (apparatus shown in Figure 3, page III-8) was postponed in order to give priority to the study of electrolyte conductance.

## 3. Material Stability,

Stability tests were started to screen out unstable electrode-electrolyte and separator-electrolyte systems from further consideration.

## C. Experimental Techniques.

### 1. Electrolyte.

In measuring the electrolytic conductance of the electrolytes a 1 kc General Radio 1650-A Impedance Bridge (Figure 4, page III-9) was used. The conductivity cells were of the dip type with cell constants of approximately  $0.1 \text{ cm}^{-1}$ . At the balance of the bridge the indicated capacitance and dissipation factor represent the properties of a R-C parallel circuit which is the equivalent circuit of the conductivity cell plus the electrolyte. This R represents the resistance of the electrolyte between the two platinum electrodes of the conductivity cell. All the specific conductance data presented in this report were calculated from such R and the proper cell constants.

In order to examine many solute-solvent systems rapidly, we chose to examine the conductances of solute-solvent mixtures at a one-molal ratio. That is, the ratio of solute to solvent was one g-mole to 1000 g. We then simply noted whether the solute did or did not dissolve completely in such a mixture. Molal solutions were used primarily because it simplified the screening work to use weight ratios in the drybox and defined the system.

The solutes used in the conductance tests were all in anhydrous form as received from the supplier's unless otherwise specified.

No solvent purification work has been performed to date because we found that the presence of traces of water in most cases exerted only minimal effect on conductance values.

Most of the conductance measurements were made in a dry argon atmosphere (Figure 4, page III-9 and Figure 5, page III-10).

The viscosity measurements of the electrolytes were performed by using Ostwald-type viscosimeters at 25°C (Figure 6, page III-11). No specific measures to isolate the sample from room air were made during the measurements.

## 2. Electrochemistry.

To evaluate qualitatively the electrochemistry of electrodes in the best electrolytes, small cells were constructed and tested. Magnesium and aluminum were tested as anode materials. A few cathode materials ( $\text{CuF}_2$ ,  $\text{NiF}_2$ , and  $\text{AgCl}$ ) were also tested. The primary objective of this work was to establish some knowledge of the open circuit potentials of the electrodes in the electrolytes, and obtain an indication of the reversibility of the electrodes as well as their ability to carry small load currents.

The potentials of the electrodes were measured with a vacuum tube voltmeter against an  $\text{AgCl}/\text{Ag}$  or metal strip reference electrode (Figure 7, page III-12). The stability of reference electrodes in nonaqueous solvents is not well established. One reason is that so many different organic systems exist which could be studied that investigators have only been able to scratch the surface in comparison to the detailed studies which have been made for aqueous reference electrodes. Therefore, it is necessary to evaluate reference electrodes in individual nonaqueous systems. Several factors which are appropriate to a good reference electrode are: (1) stable potential, (2) electrochemical reversibility at low currents, i.e., about one microampere, (3) chemical stability, and (4) the Nernst equation should be applicable. To establish an accurate value for the reference electrode potential versus the standard hydrogen electrode (SHE) requires sophisticated methods. These can be found in the literature<sup>55</sup>. However, many investigators have used metal strip reference electrodes which are merely calibrated for the stability of their potentials by comparisons of the potential of the metal strip with that of freshly prepared strips of the same metal over extended periods of time under well defined experimental conditions. The potentials of electrodes measured against such reference electrodes are arbitrary and do not define the thermodynamic electrode potential versus the SHE. However, if both half cell anode and cathode potentials are measured against such a metal strip reference electrode, the cell potential for the system is readily established as follows:

$$E_{\text{cell}} = E_{\text{cathode vs. metal strip}} - E_{\text{anode vs. metal strip}}$$

or, more explicitly:

$$\begin{aligned} E_{\text{cell}} &= (E_{\text{cathode}} - E_{\text{metal strip}}) - (E_{\text{anode}} - E_{\text{metal strip}}) \\ &= E_{\text{cathode}} - E_{\text{metal strip}} - E_{\text{anode}} + E_{\text{metal strip}} \\ &= E_{\text{cathode}} - E_{\text{anode}} \end{aligned}$$

Many other electrochemical problems exist with nonaqueous electrolytes. Anode and cathode reversibility and efficiency data are meager. Thus a great deal of research remains to be done in this area before efficient nonaqueous batteries can be built.

### 3. Material Stability.

To establish the stability of the electrode materials in the solvents (or in electrolytes), periodic visual observations were made on the samples of the material immersed in the solvent to detect gross attack of the solvent on the material. As a measure of solubility, conductance measurements were made on systems where the material was not attacked visually. Weight loss was not determined where the materials were in powder form.

In general, if the materials appeared to be stable in open atmosphere tests, they were considered stable. However, if they were unstable during the course of open atmosphere tests it was necessary to repeat the tests in a dry argon atmosphere to minimize the possible influence of moisture.

The stability of separator materials in the solvents was determined by visual observations, conductance measurements and viscosity measurements.

## D. Experimental Tests and Results.

### 1. Electrolyte.

A large number of solute-solvent systems have been tested for conductance. Some of the properties of the tested solvents are listed in Table III, page III-13.

- a. Qualitative Conductance Screening Tests. All the conductance screening results are presented in Table IV, page III-14. The solutes are arranged in alphabetical order, except for the organic solutes.

We have first selected four aprotic solvents; namely, diphenyl ether, dimethylformamide (DMF), propylene carbonate (PC), and benzonitrile (BN), and a number of solutes including simple salts, Lewis acids and Lewis acid-simple salt complexes for conductance measurements. It was found that:

1. Each of the four aprotic solvents tested yielded a range in conductance covering several orders of magnitude. This is due to the large variation in solute solubility.
2. The solvent is a dominating factor in determining the overall level of conductance. It was felt then that further study on the coordination power, the dielectric constant and the viscosity of the solvent would be necessary to explain such behavior.
3. The solutes arrange themselves with rather few exceptions in the same order of conductance in the four aprotic solvents.
4. Anion size has a direct influence on the conductance (e.g.,  $\text{PF}_6^- > \text{Cl}^- > \text{F}^-$ ). Cation influence is not apparent ( $\text{Li}^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Rb}^+$ , and  $\text{Cs}^+$ ).\*

The above findings and speculations led us to choose three more solvents; namely, N-methylformamide (NMF), tetramethylurea (TMU) and dimethyl cyanamide (DMC), as well as a large number of new solutes to test further. For the structural formulas of these solvents, see Table III, page III-13.

NMF is protic. However, it was chosen for study because of its high dielectric constant (182). TMU and DMC are both aprotic in nature. TMU was chosen to study the effect produced on the coordinating power of the solvent as a result of the substitution of the dimethylamino group for the hydrogen attached to the carbonyl group of DMF. The purpose of selecting DMC was to study the effect of the substitution of the nitrile group for the carbonyl group of DMF on the coordination power of the solvent,

---

\* In studying the ion size effect, one of the problems was that we were unable to obtain many desired solutes from our suppliers. However, we did prepare some of them by metathesis (e.g.,  $\text{KPF}_6 + \text{LiCl} = \text{KCl} + \text{LiPF}_6$  in DMF).

The new solutes we have chosen included salts with large anions (e.g.,  $\text{PF}_6^-$ ,  $\text{BF}_4^-$ ,  $\text{AsF}_6^-$  and  $\text{SbF}_6^-$ ) and we have also selected some quaternary ammonium salts because they have large cations and perhaps better solubility.

It was found that solutions in NMF (dielectric constant = 182) were only slightly more conductive than those in DMF (dielectric constant = 37), in spite of the large difference in dielectric constants. Thus it appeared that the relatively high viscosity (1.8 centipoise for NMF vs. 0.8 centipoise for DMF) might have exerted a detrimental effect on the conductance. TMU solutions (dielectric constant = 25, viscosity = 1.4 centipoise) seemed to be consistent with the above conclusion, since conductances in general were even lower than those in either NMF or DMF for comparable concentrations.

It is of interest to note that upon dissolving a quaternary ammonium salt (of  $\text{PF}_6^-$ ,  $\text{BF}_4^-$ ,  $\text{AsF}_6^-$  or  $\text{SbF}_6^-$ ) in DMC, (dielectric constant = 36, viscosity = 0.63 at 30°C), the measured conductances were about the same as the corresponding DMF (dielectric constant = 37, viscosity = 0.81 at 25°C) solutions. However, an alkali metal salt (also  $\text{PF}_6^-$ ,  $\text{BF}_4^-$ ,  $\text{AsF}_6^-$  or  $\text{SbF}_6^-$ ) solution in DMC showed somewhat lower conductance than the corresponding DMF solutions. The latter phenomenon may be due to the difference in the ability of the nitrile group of DMC and the carbonyl group of DMF to coordinate with the alkali metal cations. Since the dielectric constant and the viscosity for DMC and DMF are about the same, the similarity in behavior of the quaternaries in the two solvents implies that the resulting conductivities are not dependent upon the formation of coordination complexes between the solvents and the solutes.

Inspection of all the conductance data to date reveals that high conductance ( $>10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$ ) of aprotic solutions can be obtained by dissolving large anion solutes in DMF or quaternary ammonium salts with large anions in DMC or DMF.

- b. Quantitative Determination of Conductance-Concentration Relationships. The relationships between specific conductance and molality were determined for the following twelve systems:

1.  $\text{NaPF}_6$ -DMC
2.  $(n\text{-C}_4\text{H}_9)_2\text{NH}_2\text{AsF}_6$ -DMC (Dibutylammonium Hexafluoroarsenate)

3.  $(C_6H_5CH_2)N(CH_3)_3SbF_6$ -DMC (Benzyltrimethylammonium Hexafluoroantimonate)
4.  $CsPF_6$ -DMF
5.  $LiCl$ -DMF
6.  $KPF_6$ -DMF
7.  $NaBF_4$ -DMF
8.  $NaPF_6$ -DMF
9.  $SbCl_3$ -DMF
10.  $(O \begin{smallmatrix} \text{CH}_2\text{CH}_2 \\ \text{CH}_2\text{CH}_2 \end{smallmatrix} NH_2)PF_6$  (Morpholinium Hexafluorophosphate)
11.  $KPF_6$ -PC
12.  $KPF_6$ -TMU

In some cases the viscosity-molality relationships were also established. The results are presented in Table V, page III-35.

It was observed in general that:

1. Maximum conductance existed at a concentration of less than two-molal.
2. As the concentration exceeded that of maximum conductance, the viscosity increased drastically.

These facts imply that a two-molal concentration was too crowded for the solute ions to move freely in the solvents, and that interaction or agglomeration of solute ions might have taken place as indicated by the viscosity data.

The temperature coefficients of the conductance of  $LiCl$ -DMF solutions at two different concentrations were determined, (Figure 8, page III-48).

It can be observed that the temperature coefficient for the higher concentration solution was much higher than that of the lower concentration solution.

c. Exploratory Tests.

1. Lewis acid-ether complexes. The addition of  $BF_3$  to the commercial 47%  $BF_3$ -ether complex was performed by bubbling  $BF_3$  through the complex solution. The specific conductance was recorded as a function of time (Figure 9, page III-49). Slight increase in conductance was observed.

The additions of Lewis acids to m-dimethoxybenzene and Bis[2-(2-methoxyethoxy)ethyl] ether were also performed. Most of the Lewis acids reacted exothermically with these two ethers, and resulted in colored solutions. The conductance results are presented in Table VI, page III-50. These solutions are all too low in conductance to be used as electrolytes. Other ethers tabulated in Table II, page III-7 were not tested because of the above negative results and some technique difficulties of handling the ethers.

2. Mixed solutes. The results are presented in Table IV, page III-14. It was found that in most cases the conductances were unfavorable, although the  $\text{BeCl}_2\text{-SiF}_4\text{-DMF}$  system exhibited some synergistic effect. The order of addition of the solutes did not affect the conductance significantly.
3. Mixed solvents. We selected DMF and acetone as the first example of a mixed solvent. DMF was selected because of its good solvent power and the good conductances of some of its solutions. Acetone has a very low viscosity (0.316 centipoise) and, although not aprotic, was selected to contribute the low viscosity characteristic to the mixed solvent. The solutes tested in this mixed solvent were  $\text{KPF}_6$  and  $\text{NaPF}_6$ . The results are presented in Table VII, page III-51. It was noted that with  $\text{KPF}_6$  the use of mixtures of acetone with DMF gave higher conductivities than obtained with either solvent alone. However, this effect was observed only with  $\text{KPF}_6$  solutions. These test results demonstrate that it is feasible in certain cases to obtain solutions possessing improved conductance over that of the corresponding single solvent solutions by using the mixed solvent technique.

Another low viscosity liquid was investigated, namely, isopropyl ether (dielectric constant = 3.9, viscosity = 0.36 centipoise at 25°C). This material is completely miscible with both DMF and DMC. However,  $\text{KPF}_6$  and morpholinium hexafluorophosphate are practically insoluble in it. Upon addition of either of these salts to either isopropyl ether-DMF or isopropyl ether-DMC mixed solvents, the liquids separated into two layers.



## 2. Electrochemistry.

Eighteen electrolytes having fair to good conductance ( $>1 \times 10^{-3} \text{ ohm}^{-1} \text{ cm}^{-1}$ ) were used. The anode material was magnesium strip in all cases. Cadmium, nickel, silver-silver chloride, nickel-nickel fluoride and copper-cupric fluoride were used either as the counter electrodes or the reference electrodes. The results are presented in Table VIII, page III-54.

Poor or unstable open circuit potentials were observed for most of the systems tested. However, some of the tested systems may be promising:

- a. The charging of Mg in LiCl-DMF resulted in an open circuit potential of about 2.8 volts against a nickel strip ( $E^0$  for  $2\text{Li} + \text{NiCl}_2 = 2\text{LiCl} + \text{Ni}$  is 2.57 volts). The prospect is that Li could be used as the anode in the LiCl-DMF electrolyte.
- b. Mg and Ag-AgCl had a stable open circuit potential of about 2.0 volts in  $\text{BCl}_3$ -PC electrolyte. It is observed that water might have been present in the electrolyte. However, the best obtained open circuit potential of this electrode couple in aqueous electrolyte is only 1.7 volts.
- c. An abnormally high stable open circuit potential of 3.1 volts was obtained for the Mg and Ni-NiF<sub>2</sub> couple in KPF<sub>6</sub>-PC electrolyte after charging. Figure 10, page III-62, shows the cell voltage as a function of time through the test. These results indicate that potassium might have been plated out on Mg and have functioned as the active anode material.

It was learned from these qualitative electrochemical tests that extreme care in reduction of the moisture content in the cell systems is necessary to obtain the best results. For example, high open circuit potential was never achieved for system "c" described above when a moisture contaminated KPF<sub>6</sub>-PC solution was used.

## 3. Material Stability.

The following electrode materials were tested in the solvents DMF, PC and BN and some of their solutions:

Mg, Al, Co, Cu, Ni, NiCl<sub>2</sub>, NiF<sub>2</sub>, CuF<sub>2</sub>, CoF<sub>3</sub>

The results are presented in Table IX, page III-63. All those materials appear to be reasonably stable in the solvents and the solutes except  $\text{NiCl}_2$  which was too soluble in DMF to be used as cathode material in DMF solutions.

The tested separator materials included dacron, cotton and polypropylene. The results (Table X, page III-70) show that most of the polypropylene and dacron materials tested were stable in PC and DMF. Cotton seemed to be attacked by DMF.

## II. CONCLUSIONS AND RECOMMENDATIONS

## II. CONCLUSIONS AND RECOMMENDATIONS

### A. Conclusions

We have investigated the following in this contract period:

1. Six (6) aprotic solvents. Of these, DMF and DMC solutions show greatest promise for electrolytes.
2. Seventy-two (72) solutes. Of these, twenty-two (22) show promise as the ionic species for electrolytes. They are:

- (1)  $\text{KPF}_6$
- (2)  $\text{LiCl}$
- (3)  $\text{AlCl}_3$
- (4)  $(n\text{-C}_4\text{H}_9)\text{NI}$  (Tetrabutylammonium Iodide)
- (5)  $\text{CsPF}_6$
- (6)  $\text{NaPF}_6$
- (7)  $\text{LiPF}_6$
- (8)  $\text{FeCl}_3$
- (9)  $\text{MgCl}_2$
- (10)  $\text{NaBF}_4$
- (11)  $\text{KAsF}_6$
- (12)  $\text{NaSbF}_6$
- (13)  $\text{NH}_4\text{SO}_3\text{F}$
- (14)  $(n\text{-C}_3\text{H}_7)_4\text{NBF}_4$  (Tetrapropylammonium Tetrafluoborate)
- (15)  $(n\text{-C}_4\text{H}_9)_2\text{NH}_2\text{AsF}_6$  (Dibutylammonium Hexafluoroarsenate)
- (16)  $(n\text{-C}_3\text{H}_7)_3\text{NHAsF}_6$  (Tripropylammonium Hexafluoroarsenate)
- (17)  $(\text{C}_6\text{H}_5\text{CH}_2)\text{N}(\text{CH}_3)_3\text{SbF}_6$  (Benzyltrimethylammonium Hexafluoroantimonate)
- (18)  $(\text{CH}_3)_4\text{NPF}_6$  (Tetramethylammonium Hexafluorophosphate)
- (19)  $(\text{C}_6\text{H}_5)\text{N}(\text{CH}_3)_3\text{PF}_6$  (Phenyltrimethylammonium Hexafluorophosphate)
- (20)  $(4\text{-C}_{12}\text{H}_{25}\text{C}_6\text{H}_4\text{CH}_2)\text{N}(\text{CH}_3)_3\text{PF}_6$  (4-Dodecylbenzyltrimethylammonium Hexafluorophosphate)
- (21)  $(n\text{-C}_3\text{H}_7)_4\text{NPF}_6$  (Tetrapropylammonium Hexafluorophosphate)
- (22)  $(\text{O} \begin{array}{c} \text{CH}_2\text{CH}_2 \\ \diagup \quad \diagdown \\ \text{CH}_2\text{CH}_2 \end{array} \text{NH}_2)\text{PF}_6$  (Morpholinium Hexafluorophosphate)

The tested combinations of the above solutes and solvents yielded thirty-four (34) solutions with conductances in excess of  $1 \times 10^{-2}$  ohm<sup>-1</sup> cm<sup>-1</sup> at either one-molal concentrations or less. They are:

DMF		
	Solute (From Table Above)	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )
1.	(1)	.0237
2.	(4)	.0106
3.	(5)	.0240
4.	(6)	.0222
5.	(7)	.0206
6.	(8)	.0121
7.	(9)	.0100
8.	(10)	.0204
9.	(11)	.0241
10.	(12)	.0230
11.	(13)	.0215
12.	(14)	.0186
13.	(15)	.0227
14.	(16)	.0216
15.	(17)	.0198
16.	(18)	.0109
17.	(19)	.0208
18.	(20)	.0115
19.	(21)	.0205
20.	(22)	.0257

DMC		
21.	(1)	.0149
22.	(11)	.0181
23.	(6)	.0204
24.	(12)	.0237
25.	(14)	.0186
26.	(15)	.0228
27.	(16)	.0210
28.	(17)	.0194
29.	(20)	.0101
30.	(21)	.0216
31.	(22)	.0239

NMF		
32.	(2)	.0109
33.	(3)	.0100
34.	(4)	.0117

While the study is by no means complete, the following tentative generalizations may be made:

1. The solvent should preferably have a dielectric constant of around 20 or higher, and a viscosity of less than 3 centipoises at room temperature. Higher dielectric constant and lower viscosity will contribute to improved conductivity.
2. The ability of the solvent to form complexes with the ionic species of the solute contributes to the dissociation into ions.
3. Most of the systems investigated showed maximum conductances at solute concentrations in the range of 1 to 2 molal, in contrast to aqueous systems which show maxima at 3 to 6 molal.
4. Solution viscosity increases slowly with solute concentration to the conductance maximum, and thereafter climbs with greatly increased rapidity.
5. Conductance maxima in general appeared at higher solute concentrations in solvents of lower molecular weight. This is probably because the availability of noncoordinated solvent molecules (for a given solute concentration) goes up as molecular weight (or perhaps more precisely, molecular volume) goes down. Thus a higher concentration of solute can be achieved before the onset of excessive viscosity increases.
6. The systems investigated most intensively to date have been based on solvents whose ligand groups are capable of acting as electron donors rather than as electron acceptors. We would anticipate the formation of solvated complexes with solute cations and not with solute anions, therefore. In such systems solute solubility and solution conductance have appeared highly dependent upon anion size, with larger size being beneficial -- presumably through reduction in charge density.
7. Increasing cation size in these systems also is somewhat beneficial although not so striking as in the case of the anions. The conductance of one-molal solutions of the alkali hexafluorophosphates, for instance, increases slightly from Li through to Cs. The solubility of the quaternary ammonium salts, (which may not coordinate with the solvents), goes up with increasing cation size.
8. Cation size can perhaps become excessive. The conductance of one-molal p-dodecylbenzyl trimethylammonium hexafluorophosphate is only about half that of other quaternaries with comparable solubility. Viscosities of solutions of this salt are significantly higher than those of other salts of smaller ion size.

B. Recommendations.

For the continuation of our present work toward obtaining 200 watt-hours per pound batteries, we feel that the following studies are necessary:

1. The investigation toward obtaining nonaqueous electrolytes possessing conductance approaching that in the aqueous range should be continued by studying the coordination complexes more thoroughly. The possibility of using low melting ionic salts should also be considered.
2. Electrochemical kinetics studies should be performed using electrode couples possessing theoretical energy densities in excess of 500 watt-hours per pound, together with aprotic nonaqueous electrolytes. Properties such as activation polarization should be examined in order to achieve more understanding of the limitations on electrode behavior. Special attention should be paid to the purity of the materials used, with particular emphasis on freedom from water.
3. More extensive study on the stability of low density separator and case materials in the electrolytes should be made.
4. Systems possessing favorable electrochemical characteristics and stability should be assembled to study the performance of prototype batteries. Attention will be given to electrode design and construction to minimize the loss from "dead weight."

### III. FIGURES AND TABLES



TABLE I. THEORETICAL PROPERTIES OF ELECTRODE COUPLES

Code Number	Reaction		Equivalent Weight (g/g-equiv.)	$-\Delta F^\circ$ of Reaction (kcal/g-mole)	$E^\circ$ Volts	Energy Density (watt-hours/lb.)
110	Mg + CuO	= MgO + Cu	52.0	105.73	2.30	538
113	Mg + CuF <sub>2</sub>	= MgF <sub>2</sub> + Cu	62.7	134.8	2.92	566
116	Mg + CuCl <sub>2</sub>	= MgCl <sub>2</sub> + Cu	79.3	99.57	2.16	332
	Mg + CuSO <sub>4</sub>	= MgSO <sub>4</sub> + Cu	91.8	122.3	2.66	352
120	2Mg + NiO <sub>2</sub>	= 2MgO + Ni	34.9	224.76	2.44	850
121	Mg + NiO	= MgO + Ni	49.5	84.43	1.83	449
123	Mg + NiF <sub>2</sub>	= MgF <sub>2</sub> + Ni	60.4	102	2.21	445
126	Mg + NiCl <sub>2</sub>	= MgCl <sub>2</sub> + Ni	77.0	76.47	1.66	262
130	Mg + AgO	= MgO + Ag	74.2	138.73	2.98	491
131	Mg + Ag <sub>2</sub> O	= MgO + 2Ag	127.7	133.53	2.90	276
133	Mg + AgF <sub>2</sub>	= MgF <sub>2</sub> + Ag	85.1	210	4.55	650
134	Mg + 2AgF	= MgF <sub>2</sub> + 2Ag	138.3	162.4	3.53	310
140	4Mg + Co <sub>3</sub> O <sub>4</sub>	= 4MgO + 3Co	42.1	365.12	1.98	572
141	Mg + CoO	= MgO + Co	49.4	87.13	1.89	465
143	3Mg + 2CoF <sub>3</sub>	= 3MgF <sub>2</sub> + 2Co	51.0	416.4	3.02	720
144	Mg + CoF <sub>2</sub>	= MgF <sub>2</sub> + Co	60.5	104.35	2.26	454

TABLE I. THEORETICAL PROPERTIES OF ELECTRODE COUPLES (Continued)

Code Number	Reaction	Equivalent Weight (g/g-equiv.)	$-\Delta F^0$ of Reaction (kcal/g-mole)	$E^0$ Volts	Energy Density (watt-hours/lb.)
145	$\text{Mg} + 2\text{CoF}_3 = \text{MgF}_2 + 2\text{CoF}_2$	128	207.7	4.50	427
154	$3\text{Mg} + 2\text{AsF}_3(1) = 3\text{MgF}_2 + 2\text{As}$	56.13	367.8	2.56	575
200	$2\text{Li} + \frac{1}{2}\text{O}_2 = \text{Li}_2\text{O}$	14.95	133.9	2.91	2365
210	$2\text{Li} + \text{CuO} = \text{Li}_2\text{O} + \text{Cu}$	46.5	103.5	2.25	587
211	$2\text{Li} + 2\text{CuO} = \text{Li}_2\text{O} + \text{Cu}_2\text{O}$	86.5	108.1	2.34	329
213	$2\text{Li} + \text{CuF}_2 = 2\text{LiF} + \text{Cu}$	57.6	163.2	3.55	749
216	$2\text{Li} + \text{CuCl}_2 = 2\text{LiCl} + \text{Cu}$	74.2	141.4	3.07	503
217	$\text{Li} + \text{CuCl}_2 = \text{LiCl} + \text{CuCl}$	141.6	77.9	3.38	290
223	$2\text{Li} + \text{NiF}_2 = 2\text{LiF} + \text{Ni}$	55.5	130.4	2.83	620
226	$2\text{Li} + \text{NiCl}_2 = 2\text{LiCl} + \text{Ni}$	71.5	118.3	2.57	437
233	$2\text{Li} + \text{AgF}_2 = 2\text{LiF} + \text{Ag}$	79.9	238	5.16	786
243	$3\text{Li} + \text{CoF}_3 = 3\text{LiF} + \text{Co}$	45.8	250.8	3.64	965
244	$2\text{Li} + \text{CoF}_2 = 2\text{LiF} + \text{Co}$	55.3	132.75	2.88	633
245	$\text{Li} + \text{CoF}_3 = \text{LiF} + \text{CoF}_2$	122.5	118.05	5.14	510
254	$3\text{Li} + \text{AsF}_3(1) = 3\text{LiF} + \text{As}$	50.9	225.9	3.27	780
257	$3\text{Li} + \text{AsCl}_3(1) = 3\text{LiCl} + \text{As}$	67.4	213.6	3.09	557

TABLE I. THEORETICAL PROPERTIES OF ELECTRODE COUPLES (Continued)

Code Number	Reaction	Equivalent Weight (g/g-equiv.)	$-\Delta F^0$ of Reaction (kcal/g-mole)	$E^0$ Volts	Energy Density (watt-hours/lb.)
264	$3\text{Li} + \text{SbF}_3 = 3\text{LiF} + \text{Sb}$	66.53	215	3.11	568
310	$2\text{Al} + 3\text{CuO} = \text{Al}_2\text{O}_3 + 3\text{Cu}$	48.7	282.57	2.06	515
313	$2\text{Al} + 3\text{CuF}_2 = 2\text{AlF}_3 + 3\text{Cu}$	59.8	240	1.735	353
330	$2\text{Al} + 3\text{AgO} = \text{Al}_2\text{O}_3 + 3\text{Ag}$	70.9	384.57	2.78	477
400	$\text{Ca} + \frac{1}{2}\text{O}_2 = \text{CaO}$	28.1	144.4	3.14	1360
410	$\text{Ca} + \text{CuO} = \text{CaO} + \text{Cu}$	59.7	114	2.47	503
413	$\text{Ca} + \text{CuF}_2 = \text{CaF}_2 + \text{Cu}$	70.7	161.7	3.51	604
416	$\text{Ca} + \text{CuCl}_2 = \text{CaCl}_2 + \text{Cu}$	87.3	137.3	2.98	415
500	$\text{Be} + \frac{1}{2}\text{O}_2 = \text{BeO}$	12.44	139	3.02	2930
510	$\text{Be} + \text{CuO} = \text{BeO} + \text{Cu}$	44.3	108.6	2.36	648
513	$\text{Be} + \text{CuF}_2 = \text{BeF}_2 + \text{Cu}$	55.2	101	2.19	482
516	$\text{Be} + \text{CuCl}_2 = \text{BeCl}_2 + \text{Cu}$	72.0	69.8	1.515	256
544	$\text{Be} + \text{CoF}_2 = \text{BeF}_2 + \text{Co}$	53.0	70.55	1.53	351
600	$2\text{Na} + \frac{1}{2}\text{O}_2 = \text{Na}_2\text{O}$	31.0	90	1.95	764
610	$2\text{Na} + \text{CuO} = \text{Na}_2\text{O} + \text{Cu}$	62.8	59.6	1.28	248
613	$2\text{Na} + \text{CuF}_2 = 2\text{NaF} + \text{Cu}$	73.7	142.6	3.09	510

TABLE I. THEORETICAL PROPERTIES OF ELECTRODE COUPLES (Continued)

<u>Code Number</u>	<u>Reaction</u>	<u>Equivalent Weight (g/g-equiv.)</u>	<u><math>-\Delta F^0</math> of Reaction (kcal/g-mole)</u>	<u><math>F^0</math> Volts</u>	<u>Energy Density (watt-hours/lb.)</u>
616	$2\text{Na} + \text{CuCl}_2 = 2\text{NaCl} + \text{Cu}$	90.1	141.6	3.07	414
623	$2\text{Na} + \text{NiF}_2 = 2\text{NaF} + \text{Ni}$	71.34	109.8	2.38	405
700	$2\text{B} + 3/2 \text{O}_2 = \text{B}_2\text{O}_3$	11.6	283	2.04	2130
710	$2\text{B} + 3\text{CuO} = \text{B}_2\text{O}_3 + 3\text{Cu}$	43.2	191.8	1.385	390
713	$2\text{B} + 3\text{CuF}_2 = 2\text{BF}_3 + \text{Cu}$	54.3	174.6	1.26	Too Low
716	$2\text{B} + 3\text{CuCl}_2 = 2\text{BCl}_3 + 3\text{Cu}$	71.0	55.8	0.403	Too Low

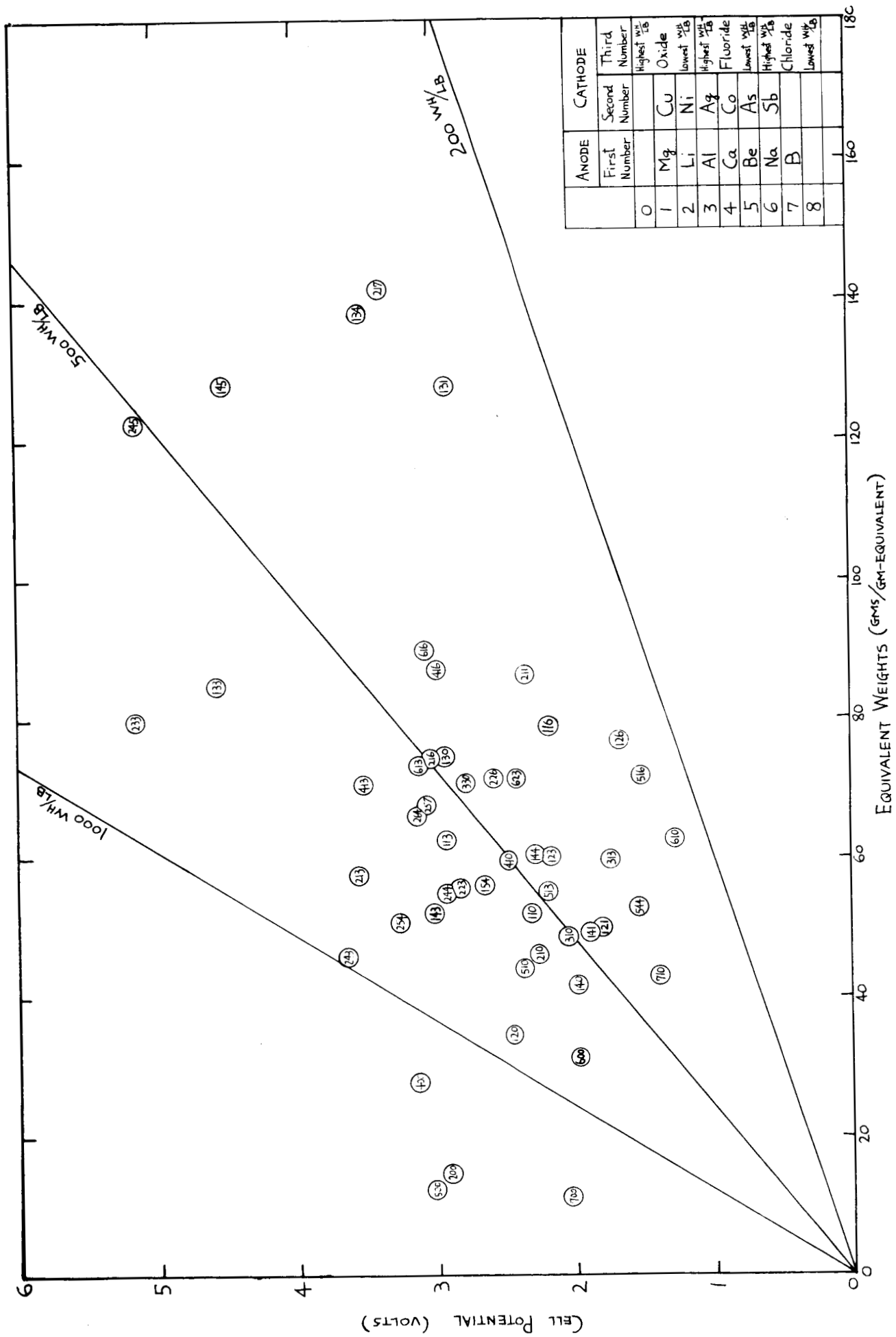
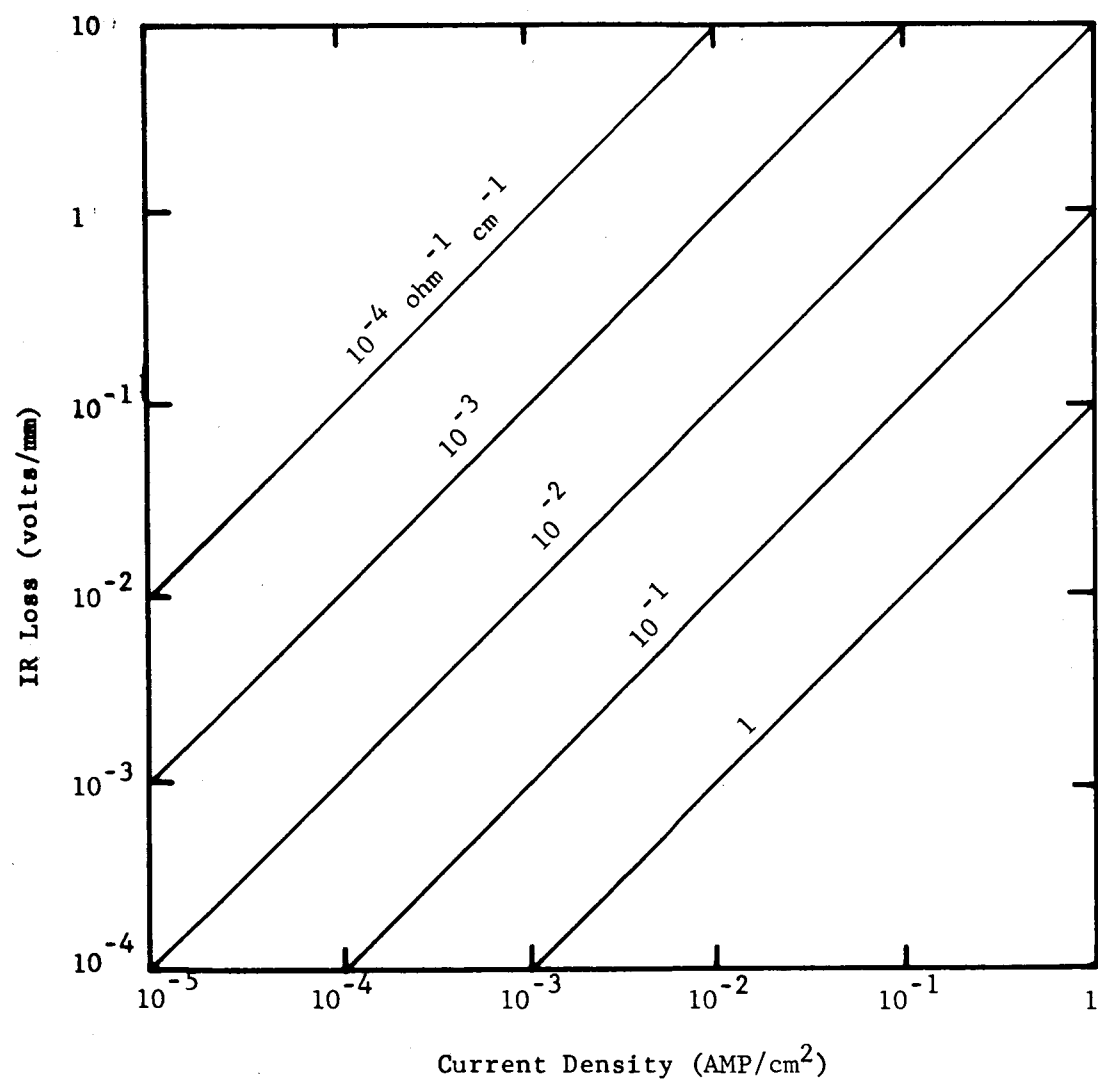



FIGURE 1 : CELL POTENTIAL, EQUIVALENT WEIGHTS AND ENERGY DENSITY RELATIONSHIP



**FIGURE 2.** EFFECT OF CONDUCTANCE OF ELECTROLYTE  
ON IR LOSS

TABLE II. SERIES OF ETHERS FOR COORDINATION STUDIES

1) Diethylether	$\text{CH}_3\text{CH}_2-\text{O}-\text{CH}_2\text{CH}_3$
2) 1, 2-dimethoxyethane	$\text{CH}_3-\text{O}-\text{CH}_2-\text{CH}_2-\text{O}-\text{CH}_3$
3) m-dimethoxybenzene	
4) Bis(2-ethoxyethyl)ether	$\text{CH}_3\text{CH}_2-\text{O}-\text{CH}_2\text{CH}_2-\text{O}-\text{CH}_2\text{CH}_2-\text{O}-\text{CH}_2\text{CH}_3$
5) 1, 2-Bis(methoxyethoxy)ethane	$\text{CH}_3-\text{O}-\text{CH}_2\text{CH}_2-\text{O}-\text{CH}_2\text{CH}_2-\text{O}-\text{CH}_2\text{CH}_2-\text{O}-\text{CH}_3$
6) Bis[2-(2-methoxyethoxy)ethyl] ether	$\text{CH}_3-\text{O}-\text{CH}_2\text{CH}_2-\text{O}-\text{CH}_2\text{CH}_2-\text{O}-\text{CH}_2\text{CH}_2-\text{O}-$ $\text{CH}_2\text{CH}_2-\text{O}-\text{CH}_3$

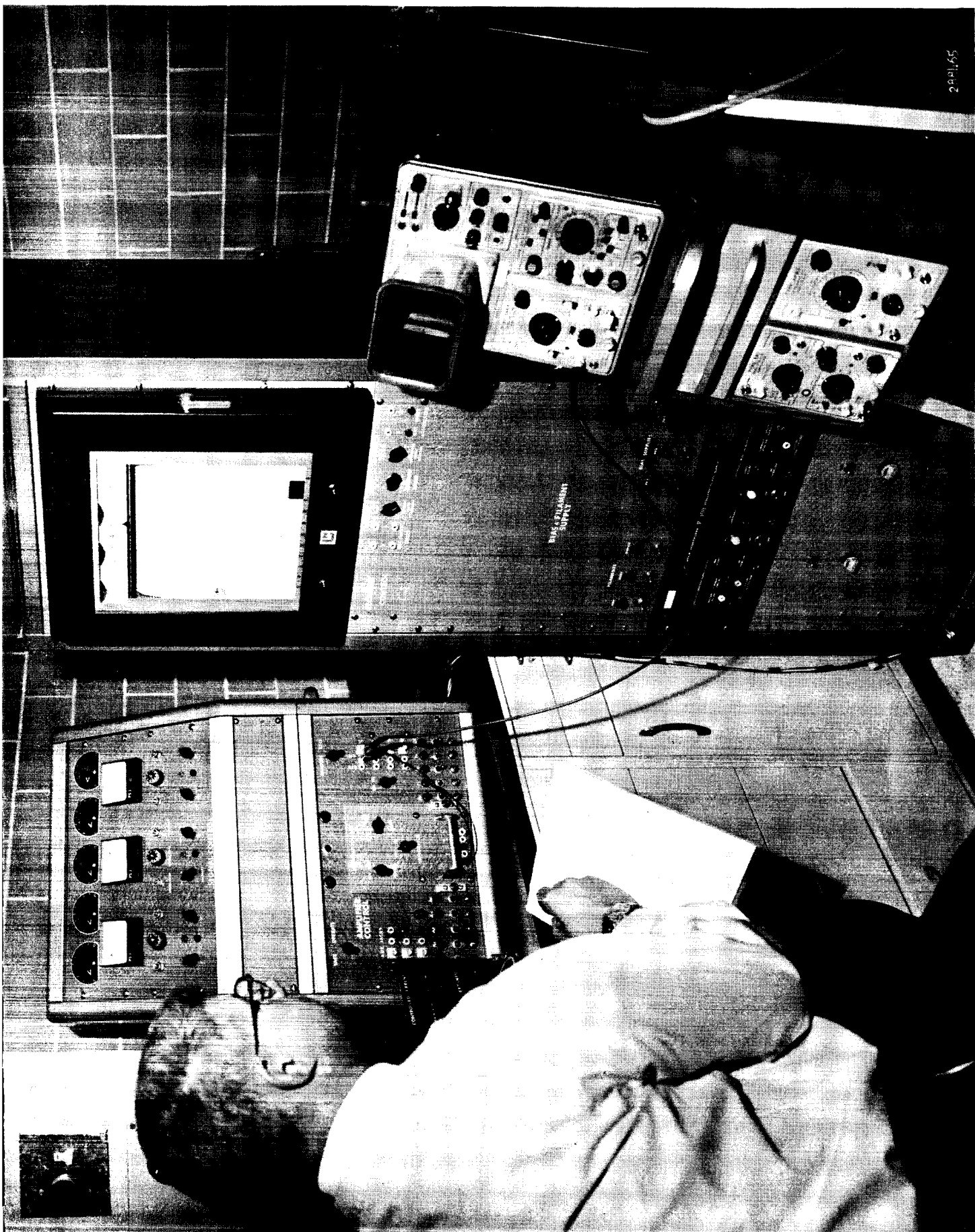


FIGURE 3. ELECTROCHEMICAL KINETICS APPARATUS



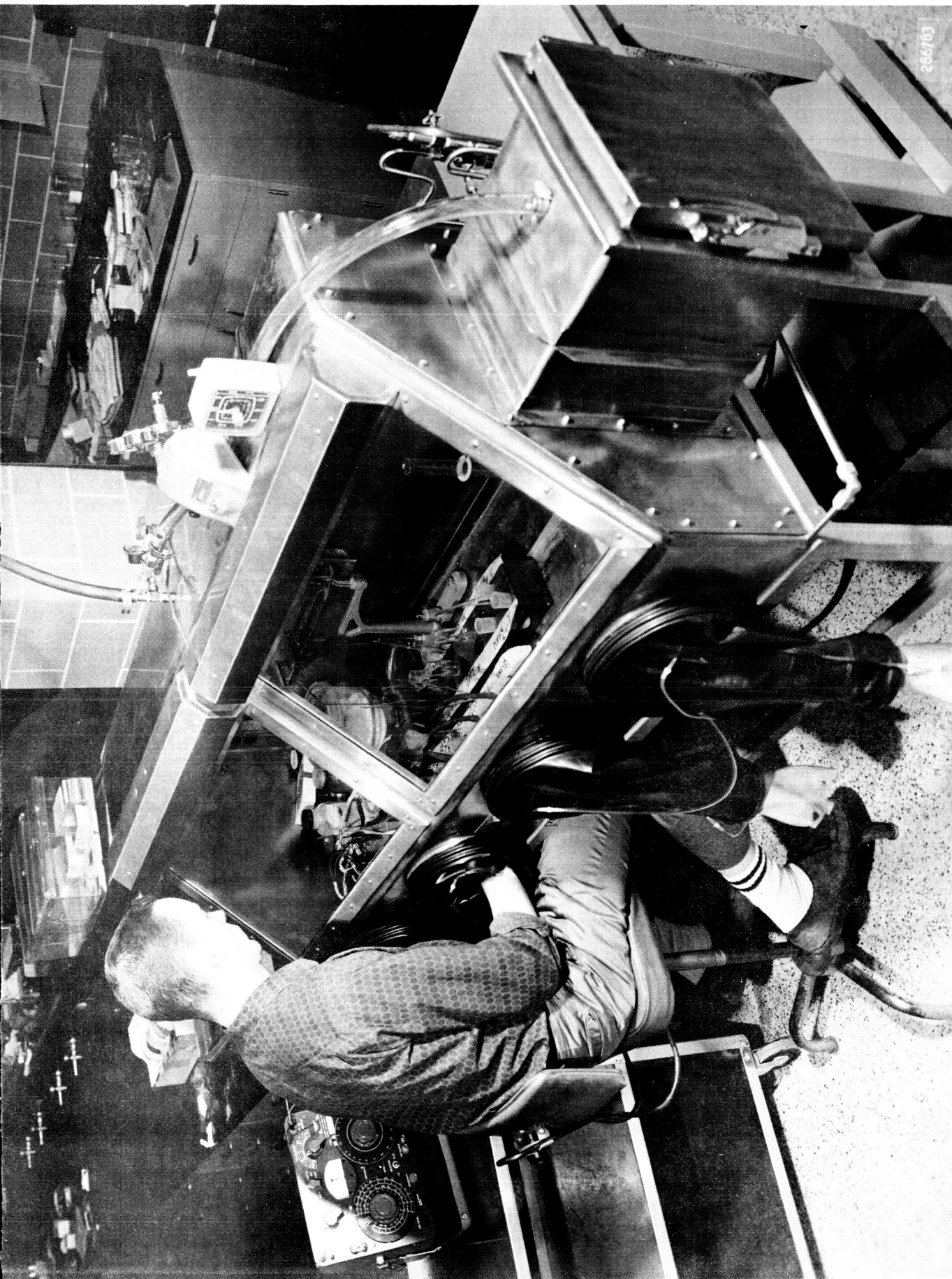


FIGURE 4. DRYBOX (ARGON ATMOSPHERE)

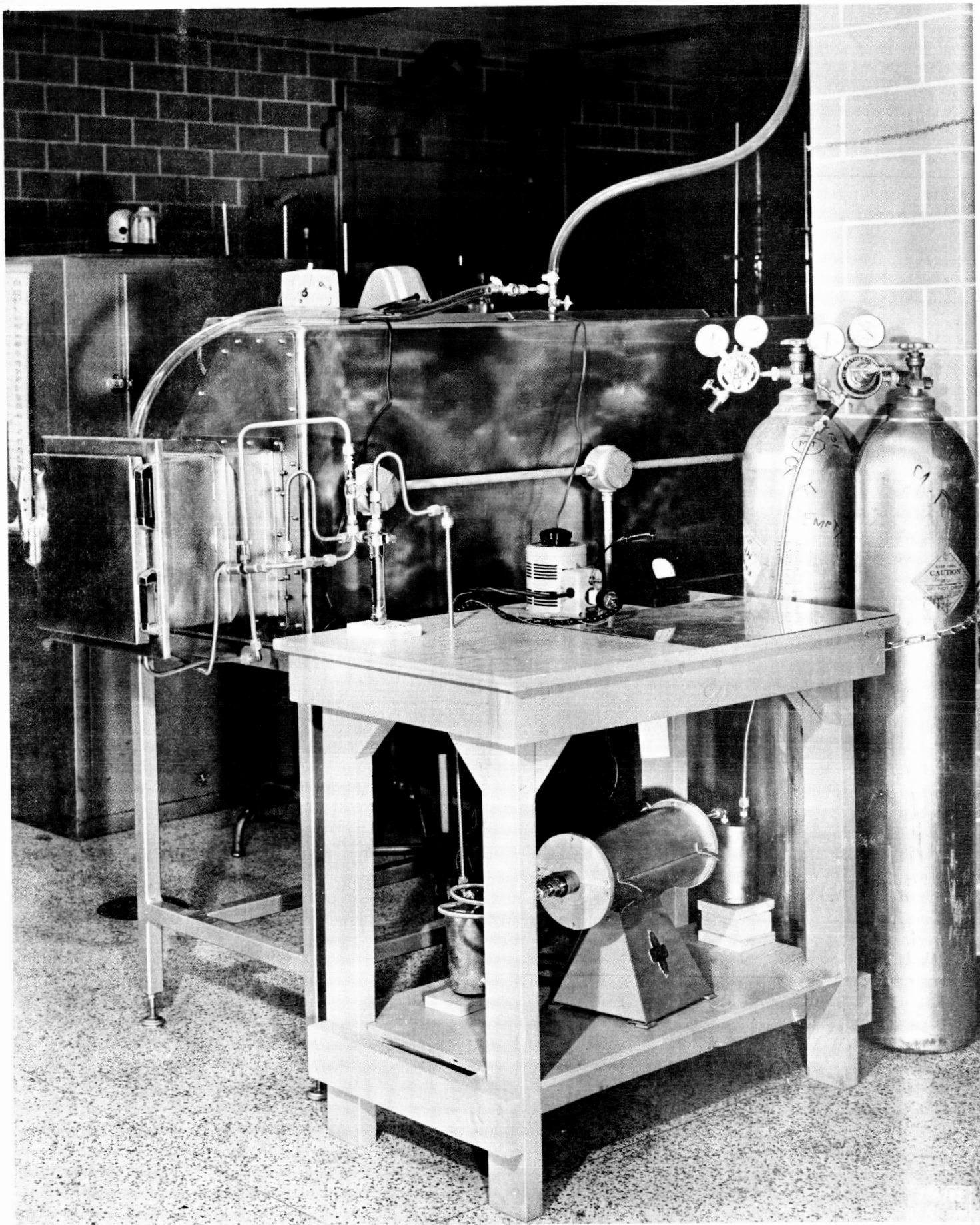


FIGURE 5. DRYBOX (INERT GAS PURIFICATION AND FEED LINES AND VALVES)





FIGURE 6. VISCOSITY APPARATUS

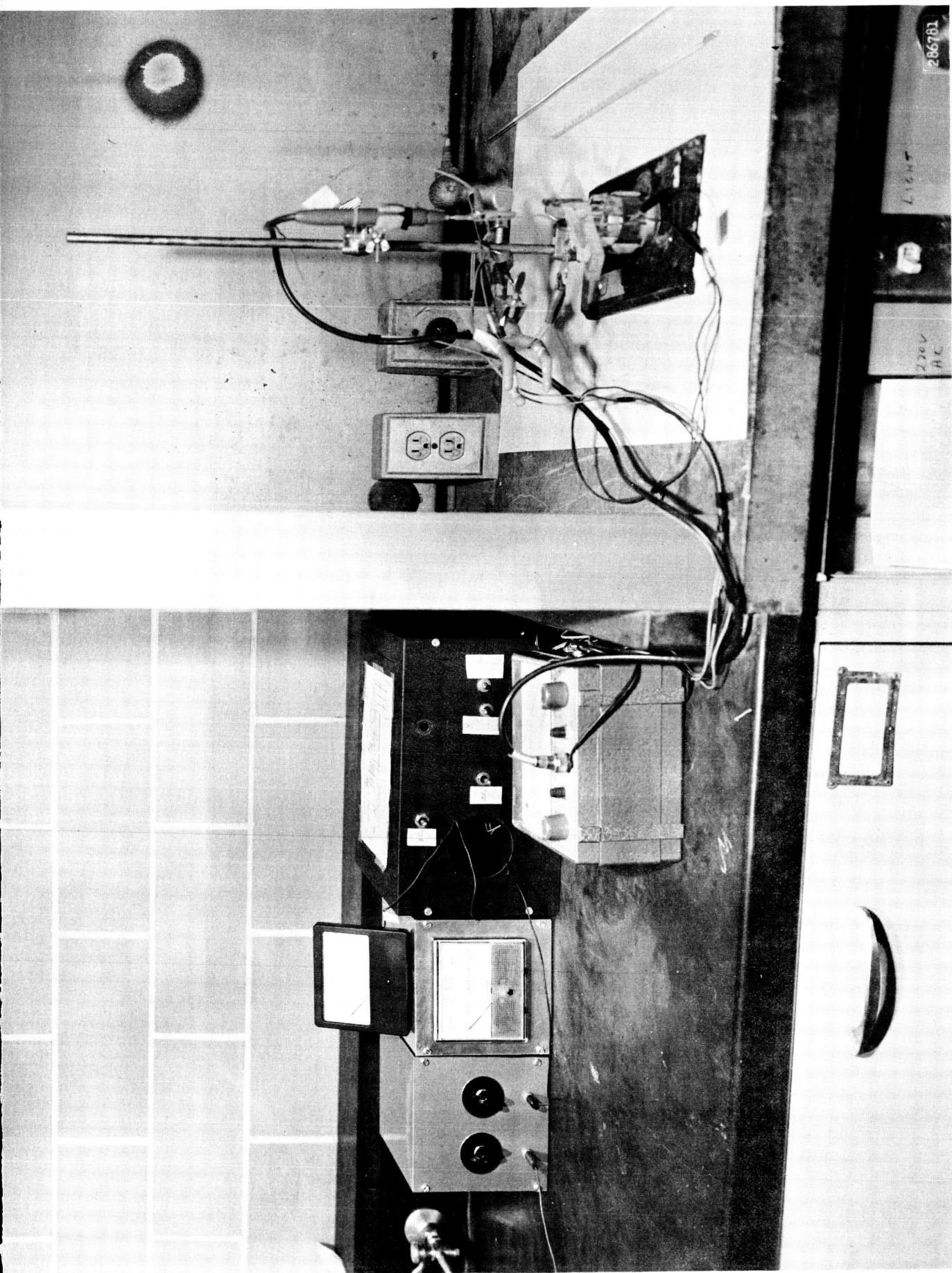
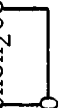


FIGURE 7. ELECTROCHEMICAL TEST APPARATUS (OPEN CIRCUIT POTENTIALS)

TABLE III. PROPERTIES OF SOLVENTS

Mol. Wt.	M.P. (°C)	B.P. (°C)	Lit. Ref.	Density (g/ml)	Temp (°C)	Lit. Ref.	Vis. (c.p.)	Temp (°C)	Lit. Ref.	Dielec. Const.	Temp (°C)	Lit. Ref.
a. <u>Acetone</u>												
CH <sub>3</sub> COCH <sub>3</sub>	58.08	-95	51	0.792	20	51	0.316	25	51	20.7	25	83
b. <u>Benzonitrile</u>												
C <sub>6</sub> H <sub>5</sub> CN	103.12	-13	51	1.0102	15	51	1.24	25	51	25.2	25	83
c. <u>Dimethyl Cyanamide</u>												
(CH <sub>3</sub> ) <sub>2</sub> NCN	70.1	-41	1	0.8768	30	1	0.633	30	1	36	R.T.	*
d. <u>Dimethylformamide</u>												
(CH <sub>3</sub> ) <sub>2</sub> NCHO	73.09	-61	31	0.9445	25	31	0.802	25	31	36.7	25	31
				0.945	25	*	0.813	25	*			
e. <u>Diphenylether</u>												
(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> O	170.20	28	51	---	--	--	---	--	--	3.65	30	83
f. <u>N-methylformamide</u>												
CH <sub>3</sub> NHCHO	59.07	--	180-185 #	0.9976	25	99	1.65	25	99	182.4	25	99
				1.0175	25	*	1.88	25	*			
g. <u>Propylene Carbonate</u>												
CH <sub>3</sub> CHCH <sub>2</sub> OCO 	102.09	-49.2	241.7	1.2057	20	58	2.84	20	58	69.0	23	58
				1.201	25	*	2.52	25	*			
h. <u>Tetramethylurea</u>												
[(CH <sub>3</sub> ) <sub>2</sub> N] <sub>2</sub> CO	116.16	--	177	0.972	15	51	1.41	25	*	25	R.T.	*
				0.964	25	*						

\* Measured Values

R.T. Room Temperature

# Aldrich Chemical Company, Inc.

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS

	<u>Page No.</u>
a. Specific Conductance of Benzonitrile Solutions . . . . .	III-15, III-16
b. Specific Conductance of Dimethyl Cyanamide Solutions . . . . .	III-17, III-18
c. Specific Conductance of Dimethylformamide Solutions . . . . .	III-19 - III-25
d. Specific Conductance of Diphenylether Solutions . . . . .	III-26
e. Specific Conductance of N-Methylformamide Solutions . . . . .	III-27
f. Specific Conductance of Propylene Carbonate Solutions . . . . .	III-28 - III-32
g. Specific Conductance of Tetramethylurea Solutions . . . . .	III-33, III-34

The following notes on concentration of solutions and experimental conditions are referred to by number throughout Table IV.

- (1) Solution was saturated. Test was made in open atmosphere.
- (2) Gaseous solute was bubbled through the solvent for ten minutes. Conductance was measured one hour afterward. Test was made in open atmosphere.
- (3) Solute formed solid reaction product with the solvent. Conductance was measured after redissolving some of the solid product. Test was made in open atmosphere.
- (4) Solute (or each solute) to solvent ratio was 1 g-mole to 1 kg. Solution was apparently saturated. Test was made in a dry argon atmosphere.
- (5) One molal solution. Test was made in a dry argon atmosphere.
- (6) Solute at concentration of maximum conductance. Test was made in a dry argon atmosphere.
- (7) One gaseous solute and one solid solute were added to the solvent in the order shown. The solid solute to solvent ratio was 1 g-mole to 1 kg. The gaseous solute was bubbled through the solution (or solvent) until a maximum conductance was obtained. Test was made in a dry argon atmosphere.
- (8) Approximately a one molal solution. Test was made in open atmosphere.
- (9) Solution was saturated. Test was made in  $P_2O_5$  desiccated atmosphere.
- (\*) Room temperature. This is about 25-30°C.

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS

## a. Specific Conductance of Benzonitrile Solutions

Solute	Experimental Conditions	Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Visual Observations		
			Solution	Solid	Others
Blank		$1.2 \times 10^{-7}$ (25°C)			
AlCl <sub>3</sub>	(1)	$1.63 \times 10^{-3}$ (*)	Brown	Orange	Exothermic
	(6)	$3.85 \times 10^{-3}$ (25°C)	Brown	---	---
AlF <sub>3</sub>	(1)	$1.51 \times 10^{-7}$ (24.5°C)	---	---	---
ECI <sub>3</sub> (g)	(2)	$4.43 \times 10^{-5}$ (*)	Red brown	---	Exothermic
BF <sub>3</sub> (g)	(2)	$1.20 \times 10^{-4}$ (27.5°C)	---	---	Exothermic
BeCl <sub>2</sub>	(9)	$5.80 \times 10^{-5}$ (*)	Brown	---	---
BeCl <sub>2</sub> +KCl	(9)	$5.45 \times 10^{-5}$ (*)	Brown	---	---
BeCl <sub>2</sub> +LiCl	(9)	$7.18 \times 10^{-5}$ (*)	Tan	---	---
BeCl <sub>2</sub> +MgCl <sub>2</sub>	(9)	$6.97 \times 10^{-5}$ (*)	Light tan	---	---
BeF <sub>2</sub>	(9)	$1.15 \times 10^{-6}$ (*)	Clear	---	---
BeF <sub>2</sub> +KF	(9)	$1.06 \times 10^{-6}$ (*)	Clear	---	---
BeF <sub>2</sub> +LiF	(9)	$7.87 \times 10^{-7}$ (*)	Clear	---	---
BeF <sub>2</sub> +MgF <sub>2</sub>	(9)	$1.19 \times 10^{-6}$ (*)	Clear	---	---
CaF <sub>2</sub>	(1)	$1.09 \times 10^{-7}$ (*)	---	---	---
K <sub>2</sub> CO <sub>3</sub>	(1)	$2.09 \times 10^{-6}$ (*)	---	---	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)a. Specific Conductance of Benzonitrile Solutions (Continued)

<u>Solute</u>	<u>Experimental Conditions</u>	<u>Specific Conductance (ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Visual Observations</u>		
			<u>Solution</u>	<u>Solid</u>	<u>Others</u>
KF	(9)	$1.12 \times 10^{-6}$ (*)	Clear	White	---
KI	(1)	$1.28 \times 10^{-4}$ (24°C)	---	---	---
KPF <sub>6</sub>	(9)	$3.56 \times 10^{-4}$ (*)	Clear	---	---
Li <sub>2</sub> CO <sub>3</sub>	(1)	$1.10 \times 10^{-7}$ (*)	---	---	---
LiCl	(1)	$1.40 \times 10^{-6}$ (*)	---	---	---
LiF	(1)	$1.12 \times 10^{-7}$ (*)	---	---	---
MgCl <sub>2</sub> ·6H <sub>2</sub> O (dried)	(1)	$6.55 \times 10^{-7}$ (*)	---	---	---
MgF <sub>2</sub>	(1)	$1.79 \times 10^{-7}$ (*)	---	---	---
NaBF <sub>4</sub>	(9)	$3.86 \times 10^{-5}$ (*)	Clear	---	---
NaF	(9)	$7.92 \times 10^{-7}$ (*)	Clear	---	---

(\*) Room Temperature



TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## b. Specific Conductance of Dimethyl Cyanamide Solutions

Solute	Experimental Conditions	Specific Conductance ( $\text{ohm}^{-1} \text{cm}^{-1}$ )	Visual Observations		
			Solution	Solid	Others
Blank		$4.48 \times 10^{-5}$ (31°C)			
CsCl	(4)	$7.16 \times 10^{-5}$ (34°C)	Clear	White	---
CsPF <sub>6</sub>	(4)	$6.43 \times 10^{-3}$ (29°C)	Clear	White	---
KAsF <sub>6</sub>	(5)	$1.81 \times 10^{-2}$ (28.5°C)	Clear	White	---
K <sub>2</sub> NbF <sub>7</sub>	(4)	$3.22 \times 10^{-4}$ (26°C)	Clear	White	---
KPF <sub>6</sub>	(4)	$1.49 \times 10^{-2}$ (25°C)	Clear	Slight Amount White	---
K <sub>2</sub> TaF <sub>7</sub>	(4)	$1.51 \times 10^{-4}$ (26°C)	Clear	White	---
LiCl	(4)	$1.30 \times 10^{-3}$ (34°C)	Clear	White	---
NH <sub>4</sub> SO <sub>3</sub> F	(4)	$8.93 \times 10^{-3}$ (28°C)	Clear	White	---
NaBF <sub>4</sub>	(4)	$8.10 \times 10^{-3}$ (34°C)	Clear	White	---
NaPF <sub>6</sub>	(4)	$2.04 \times 10^{-2}$ (29°C)	Turbid	Gray	---
NaSbF <sub>6</sub>	(5)	$2.37 \times 10^{-2}$ (28.5°C)	Turbid	White	---
(CH <sub>3</sub> ) <sub>4</sub> NBF <sub>4</sub> (Tetramethylammonium tetrafluoroborate)	(4)	$2.57 \times 10^{-3}$ (28°C)	Clear	Crystal	---
(n-C <sub>3</sub> H <sub>7</sub> ) <sub>4</sub> NBF <sub>4</sub> (Tetra-n-propylammonium tetrafluoroborate)	(5)	$1.86 \times 10^{-2}$ (29°C)	Gold	---	Became saturated at <1.28 m
(n-C <sub>4</sub> H <sub>9</sub> ) <sub>2</sub> NH <sub>2</sub> AsF <sub>6</sub> (Di-n-butylammonium hexafluoroarsenate)	(5)	$2.28 \times 10^{-2}$ (29°C)	Clear	---	---

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## b. Specific Conductance of Dimethyl Cyanamide Solutions (Continued)

Solute	Experimental Conditions	Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Visual Observations		
			Solution	Solid	Others
(n-C <sub>3</sub> H <sub>7</sub> ) <sub>3</sub> NHAsF <sub>6</sub> (Tri-n-propylammonium hexafluoroarsenate)	(5)	$2.10 \times 10^{-2}$ (28.5°C)	Brown	White	---
(C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> )N(CH <sub>3</sub> ) <sub>3</sub> SbF <sub>6</sub> (N-benzyl N,N-trimethylammonium hexafluoroantimonate)	(5)	$1.94 \times 10^{-2}$ (29.5°C)	Gold	---	---
(CH <sub>3</sub> ) <sub>4</sub> NPF <sub>6</sub> (Tetramethylammonium hexafluorophosphate)	(4)	$3.44 \times 10^{-3}$ (28°C)	Turbid	---	---
(4-C <sub>12</sub> H <sub>25</sub> C <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> )N(CH <sub>3</sub> ) <sub>3</sub> PF <sub>6</sub> (5) (4-dodecylbenzyltrimethylammonium hexafluorophosphate)	(5)	$1.01 \times 10^{-2}$ (30°C)	Gold	---	Over Max. Con. Already
(n-C <sub>3</sub> H <sub>7</sub> ) <sub>4</sub> NPF <sub>6</sub> (Tetra-n-propylammonium hexafluorophosphate)	(5)	$2.16 \times 10^{-2}$ (30°C)	Brown	---	---
(O $\begin{smallmatrix} \text{CH}_2\text{CH}_2 \diagup \\ \text{CH}_2\text{CH}_2 \diagdown \end{smallmatrix}$ NH <sub>2</sub> )PF <sub>6</sub> (5) (Morpholinium hexafluorophosphate)	(5)	$2.39 \times 10^{-2}$ (28.5°C)	---	---	---
(CH <sub>3</sub> ) <sub>4</sub> NBr (Tetramethylammonium bromide)	(4)	$2.43 \times 10^{-4}$ (24.5°C)	Clear	White	---
(CH <sub>3</sub> ) <sub>4</sub> NCI (Tetramethylammonium chloride)	(4)	$4.21 \times 10^{-4}$ (24.5°C)	Clear	White	---

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## c. Specific Conductance of Dimethylformamide Solutions

Solute	Experimental Conditions	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Visual Observations		
			Solution	Solid	Others
Blank		1-2 x 10 <sup>-6</sup> (25°C)			
AlCl <sub>3</sub> +LiCl	(4)	6.31 x 10 <sup>-3</sup> (28°C)	Clear	Cream	Exothermic
AlCl <sub>3</sub> ·DMF	(3)	4.55 x 10 <sup>-3</sup> (24°C)	---	---	Vigorous reaction
AlCl <sub>3</sub> ·NMF	(3)	3.62 x 10 <sup>-3</sup> (24°C)	---	---	Gas evolution
AlF <sub>3</sub>	(1)	2.24 x 10 <sup>-6</sup> (24°C)	---	---	---
BCl <sub>3</sub> (g)	(2)	1.17 x 10 <sup>-3</sup> (26.5°C)	---	---	Exothermic
BF <sub>3</sub> (g)	(2)	6.17 x 10 <sup>-3</sup> (27.5°C)	---	---	Exothermic
	(6)	9.66 x 10 <sup>-3</sup> (28°C)	Clear	---	Exothermic
BeCl <sub>2</sub>	(4)	2.10 x 10 <sup>-3</sup> (*)	Clear green	White	Exothermic
BeCl <sub>2</sub> +KCl	(4)	2.59 x 10 <sup>-3</sup> (*)	Clear green	White	---
BeCl <sub>2</sub> +LiCl	(4)	6.75 x 10 <sup>-3</sup> (*)	Clear green	White	---
BeCl <sub>2</sub> +MgCl <sub>2</sub>	(4)	8.17 x 10 <sup>-3</sup> (*)	Clear green	White	---
BeCl <sub>2</sub> +SiF <sub>4</sub>	(7)	5.72 x 10 <sup>-3</sup> (28°C)	Green	White	Exothermic
BeF <sub>2</sub>	(4)	3.49 x 10 <sup>-6</sup> (*)	Clear	White	---
BeF <sub>2</sub> +KF	(4)	5.13 x 10 <sup>-6</sup> (*)	Clear	White	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## c. Specific Conductance of Dimethylformamide Solutions (Continued)

<u>Solute</u>	<u>Experimental Conditions</u>	<u>Specific Conductance (ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Visual Observations</u>		
			<u>Solution</u>	<u>Solid</u>	<u>Others</u>
BeF <sub>2</sub> +LiF	(4)	4.77 x 10 <sup>-6</sup> (*)	Clear	White	---
BeF <sub>2</sub> +MgF <sub>2</sub>	(4)	5.02 x 10 <sup>-6</sup> (*)	Clear	White	---
CaF <sub>2</sub>	(1)	1.59 x 10 <sup>-6</sup> (*)	---	---	---
CaTiF <sub>6</sub>	(4)	2.32 x 10 <sup>-4</sup> (27.5°C)	Milky	White	---
CsCl	(4)	2.89 x 10 <sup>-4</sup> (27°C)	Clear	White	---
CsF	(4)	1.79 x 10 <sup>-4</sup> (*)	Clear	White	---
CsPF <sub>6</sub>	(5)	2.40 x 10 <sup>-2</sup> (28.5°C)	Clear	---	---
FeCl <sub>3</sub>	(5)	1.21 x 10 <sup>-2</sup> (30°C)	Brown	---	Exothermic
FeCl <sub>3</sub> +LiF	(4)	9.88 x 10 <sup>-3</sup> (26.5°C)	Brown	Yellow	Exothermic
FeF <sub>3</sub>	(4)	8.95 x 10 <sup>-5</sup> (24°C)	Clear	Brown	---
InCl <sub>3</sub>	(4)	6.34 x 10 <sup>-4</sup> (28°C)	Clear	Small Amount White	---
K <sub>2</sub> CO <sub>3</sub>	(1)	1.98 x 10 <sup>-5</sup> (*)	---	---	---
K <sub>3</sub> AlF <sub>6</sub>	(4)	7.53 x 10 <sup>-3</sup> (29°C)	Clear	White	---
KAsF <sub>6</sub>	(4)	2.41 x 10 <sup>-2</sup> (29°C)	Clear	White	---
KBF <sub>4</sub>	(4)	7.17 x 10 <sup>-3</sup> (28°C)	Clear	White	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

c. Specific Conductance of Dimethylformamide Solutions (Continued)				
Solute	Experimental Conditions	Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Solution	Visual Observations Solid Others
$\text{K}_2\text{BeF}_4$	(4)	$1.15 \times 10^{-4}$ (28°C)	Clear	White ---
KCl	(4)	$1.78 \times 10^{-4}$ (28°C)	Clear	White ---
$\text{K}_3\text{Cr}(\text{SCN})_6$	(5)	$5.75 \times 10^{-3}$ (25°C)	Opaque purple	--- Over Max. Cond. Already
KF	(4)	$4.90 \times 10^{-5}$ (*)	Clear	White ---
$\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$	(4)	$7.20 \times 10^{-6}$ (25.5°C)	Clear	Yellow ---
KI	(1) (5)	$1.94 \times 10^{-2}$ (24°C) $2.22 \times 10^{-2}$ (*)	Clear Clear	--- ---
$\text{K}_2\text{NbF}_7$	(4)	$1.45 \times 10^{-3}$ (26°C)	Clear	White ---
$\text{KPF}_6$	(5)	$2.37 \times 10^{-2}$ (27°C)	Clear	--- ---
$\text{KPF}_6 + \text{SiF}_4$	(7)	$2.24 \times 10^{-2}$ (27°C)	Clear	White Exothermic
$\text{K}_2\text{SiF}_6$	(4)	$4.14 \times 10^{-6}$ (*)	Clear	White ---
$\text{K}_2\text{TaF}_7$	(4)	$2.40 \times 10^{-4}$ (26°C)	Clear	White ---
$\text{K}_2\text{TiF}_6$	(4)	$1.14 \times 10^{-5}$ (31°C)	Clear	White ---
$\text{K}_2\text{ZrF}_6$	(4)	$5.31 \times 10^{-6}$ (27°C)	Clear	White ---
$\text{Li}_2\text{CO}_3$	(1)	$2.02 \times 10^{-6}$ (*)	---	--- ---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## c. Specific Conductance of Dimethylformamide Solutions (Continued)

Solute	Experimental Conditions	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Visual Observations		
			Solution	Solid	Others
LiCl	(1)	$2.18 \times 10^{-3}$ (*)	---	---	Exothermic
LiF	(1)	$5.64 \times 10^{-6}$ (*)	---	---	---
LiPF <sub>6</sub>	(5)	$2.06 \times 10^{-2}$ (28.5°C)	Clear	---	---
LiPF <sub>6</sub> ·H <sub>2</sub> O	(4)	$9.77 \times 10^{-4}$ (28°C)	Clear	White	---
Li <sub>2</sub> SiF <sub>6</sub>	(4)	$1.12 \times 10^{-3}$ (28°C)	Clear	White	---
LiSO <sub>3</sub> F	(4)	$1.98 \times 10^{-3}$ (28°C)	Clear	White	---
MgCl <sub>2</sub>	(4)	$1.00 \times 10^{-2}$ (28°C)	Clear	White	---
MgCl <sub>2</sub> ·6H <sub>2</sub> O (dried)	(1)	$3.86 \times 10^{-3}$ (23.5°C)	Milky clear	---	---
MgF <sub>2</sub>	(1)	$4.03 \times 10^{-6}$ (25°C)	---	---	---
	(4)	$3.55 \times 10^{-6}$ (27°C)	Clear	White	---
MgF <sub>2</sub> +SiF <sub>4</sub>	(7)	$6.18 \times 10^{-4}$ (27°C)	Clear	White	Exothermic
MgSiF <sub>6</sub>	(4)	$3.37 \times 10^{-3}$ (*)	Clear	White	---
NH <sub>4</sub> SO <sub>3</sub> F	(4)	$2.15 \times 10^{-2}$ (28°C)	Clear	White	---
Na <sub>3</sub> AlF <sub>6</sub>	(4)	$3.68 \times 10^{-5}$ (28°C)	Clear	White	---
NaAsF <sub>6</sub>	(4)	$4.46 \times 10^{-3}$ (29°C)	Milky	White	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## c. Specific Conductance of Dimethylformamide Solutions (Continued)

Solute	Experimental Conditions	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Visual Observations		
			Solution	Solid	Others
NaBF <sub>4</sub>	(5)	2.04 x 10 <sup>-2</sup> (27°C)	Clear	White	---
NaCl	(4)	3.74 x 10 <sup>-4</sup> (27°C)	Clear	White	---
NaF	(4)	3.41 x 10 <sup>-6</sup> (*)	Clear	White	---
NaPF <sub>6</sub>	(5)	2.22 x 10 <sup>-2</sup> (28°C)	Tan	Tan	Exothermic
NaSbF <sub>6</sub>	(4)	2.30 x 10 <sup>-2</sup> (31°C)	Light gray	White	---
Na <sub>2</sub> SiF <sub>6</sub>	(4)	4.76 x 10 <sup>-6</sup> (*)	Clear	White	---
Ni(PF <sub>6</sub> ) <sub>2</sub> ·H <sub>2</sub> O	(4)	4.21 x 10 <sup>-3</sup> (28°C)	Green	Green	---
RbF	(4)	2.24 x 10 <sup>-4</sup> (*)	Clear	White	---
SbCl <sub>3</sub>	(5)	1.49 x 10 <sup>-3</sup> (26°C)	Clear	---	---
SbF <sub>3</sub>	(4)	2.48 x 10 <sup>-4</sup> (25°C)	Clear	Small Amount White	---
SiF <sub>4</sub> (g)	(6)	2.45 x 10 <sup>-4</sup> (29°C)	Clear	---	Exothermic
SiF <sub>4</sub> +BeCl <sub>2</sub>	(7)	5.49 x 10 <sup>-3</sup> (28°C)	Light green	White	Exothermic
SiF <sub>4</sub> +MgF <sub>2</sub>	(7)	1.05 x 10 <sup>-3</sup> (27°C)	Clear	White	Exothermic
SiF <sub>4</sub> +RbF	(7)	6.03 x 10 <sup>-4</sup> (28°C)	Clear	White	---
SiF <sub>4</sub> +TiF <sub>4</sub>	(7)	9.36 x 10 <sup>-4</sup> (28°C)	Clear	White	Exothermic

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

c. <u>Specific Conductance of Dimethylformamide Solutions (Continued)</u>					
<u>Solute</u>	<u>Experimental Conditions</u>	<u>Specific Conductance (ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Visual Observations</u>		
			<u>Solution</u>	<u>Solid</u>	<u>Others</u>
SnF <sub>2</sub>	(4)	1.12 x 10 <sup>-4</sup> (26°C)	Clear	White	---
SnF <sub>4</sub>	(4)	8.25 x 10 <sup>-5</sup> (27°C)	Clear	Gray	---
TiF <sub>3</sub>	(4)	9.48 x 10 <sup>-5</sup> (29°C)	Brown	Brown	---
TiF <sub>4</sub>	(4)	2.69 x 10 <sup>-4</sup> (29°C)	Clear	White	---
TlF	(4)	3.99 x 10 <sup>-5</sup> (27°C)	Clear	White	---
(CH <sub>3</sub> ) <sub>4</sub> NBF <sub>4</sub> (Tetramethylammonium tetrafluoroborate)	(4)	6.89 x 10 <sup>-3</sup> (27°C)	Clear	White	---
(n-C <sub>3</sub> H <sub>7</sub> ) <sub>4</sub> NBF <sub>4</sub> (Tetra-n-propylammonium tetrafluoroborate)	(5)	1.86 x 10 <sup>-2</sup> (27°C)	Clear tan	---	---
(n-C <sub>4</sub> H <sub>9</sub> ) <sub>2</sub> NH <sub>2</sub> AsF <sub>6</sub> (Di-n-butylammonium hexafluoroarsenate)	(5)	2.27 x 10 <sup>-2</sup> (27°C)	Clear	---	---
(n-C <sub>3</sub> H <sub>7</sub> ) <sub>3</sub> NHAsF <sub>6</sub> (Tri-n-propylammonium hexafluoroarsenate)	(5)	2.16 x 10 <sup>-2</sup> (28°C)	Milky	---	---
(C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> )N(CH <sub>3</sub> ) <sub>3</sub> SbF <sub>6</sub> (N-benzyl N,N,N-trimethylammonium hexafluoroantimonate)	(5)	1.98 x 10 <sup>-2</sup> (28°C)	Tan green	---	---
[(C <sub>4</sub> H <sub>9</sub> ) <sub>2</sub> NH <sub>2</sub> ] <sub>2</sub> SiF <sub>6</sub> (Di-n-butylammonium hexafluorosilicate)	(4)	6.51 x 10 <sup>-4</sup> (28°C)	Clear	Crystal	---
(CH <sub>3</sub> ) <sub>4</sub> NPF <sub>6</sub> (Tetramethylammonium hexafluorophosphate)	(4)	1.09 x 10 <sup>-2</sup> (26°C)	Clear	White	---



TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## c. Specific Conductance of Dimethylformamide Solutions (Continued)

Solute	Experimental Conditions	Specific Conductance ( $\text{ohm}^{-1} \text{cm}^{-1}$ )	Visual Observations		
			Solution	Solid	Others
$(\text{C}_6\text{H}_5)_3\text{N}(\text{CH}_3)_3\text{PF}_6$ (N-phenyl N,N,N-trimethylammonium hexafluorophosphate)	(5)	$2.08 \times 10^{-2}$ (26.5°C)	Clear	---	---
$(4-\text{C}_{12}\text{H}_{25}\text{C}_6\text{H}_4\text{CH}_2)_3\text{N}(\text{CH}_3)_3\text{PF}_6$ (5) (N-(4-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate)	(5)	$1.15 \times 10^{-2}$ (27°C)	Yellow	---	---
$(n\text{-C}_3\text{H}_7)_4\text{NPF}_6$ (Tetra-n-propylammonium tetrafluoroborate)	(5)	$2.05 \times 10^{-2}$ (26°C)	Amber	---	---
$(\text{O}-\begin{array}{c} \text{CH}_2\text{CH}_2 \\ \diagup \quad \diagdown \\ \text{CH}_2\text{CH}_2 \end{array}-\text{NH}_2)_3\text{PF}_6$ (Morpholinium hexafluorophosphate)	(5)	$2.57 \times 10^{-2}$ (27°C)	Tan	---	---
$(\text{CH}_3)_4\text{NBr}$ (Tetramethylammonium bromide)	(4)	$5.83 \times 10^{-4}$ (24.5°C)	Clear	White	---
$(\text{CH}_3)_4\text{NCl}$ (Tetramethylammonium chloride)	(4)	$8.17 \times 10^{-4}$ (24.5°C)	Clear	White	---
$(n\text{-C}_4\text{H}_9)_4\text{NI}$ (Tetra-n-butylammonium iodide)	(5)	$1.06 \times 10^{-2}$ (28°C)	Clear	---	---

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

d. Specific Conductance of Diphenylether Solutions				
<u>Solute</u>	<u>Experimental Conditions</u>	<u>Specific Conductance (ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Solution</u>	<u>Visual Observations</u>
Blank No. 1		1.44 x 10 <sup>-8</sup> (45°C)		
Blank No. 2		4.3 x 10 <sup>-9</sup> (45°C)		
LiCl in No. 1	(1)	1.89 x 10 <sup>-8</sup> (39°C)	Clear	White ---
MgCl <sub>2</sub> in No. 1	(1)	1.87 x 10 <sup>-8</sup> (41°C)	Clear	White ---
AlCl <sub>3</sub> in No. 2	(1)	4.86 x 10 <sup>-9</sup> (37°C)	Clear	White ---
BCl <sub>3</sub> (g) in No. 2	(2)	4.61 x 10 <sup>-9</sup> (44°C)	---	---
BF <sub>3</sub> (g) in No. 2	(2)	3.81 x 10 <sup>-9</sup> (47°C)	Milky white	---

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## e. Specific Conductance of N-Methylformamide Solutions

<u>Solute</u>	<u>Experimental Conditions</u>	<u>Specific Conductance (ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Visual Observations</u>		
			<u>Solution</u>	<u>Solid</u>	<u>Others</u>
Blank No. 1		3-4 x 10 <sup>-4</sup> (*)			
Blank No. 2		5-6 x 10 <sup>-3</sup> (*)			
AlCl <sub>3</sub> ·NMF	(1)	1.00 x 10 <sup>-2</sup> (25°C)	---	---	Exothermic
AlF <sub>3</sub>	(1)	3.44 x 10 <sup>-4</sup> (25°C)	---	---	---
BF <sub>3</sub> (g)	(2)	2.69 x 10 <sup>-3</sup> (24.5°C)	---	---	Exothermic
KI	(8)	1.36 x 10 <sup>-2</sup> (24°C)	---	---	---
LiCl	(8)	1.09 x 10 <sup>-2</sup> (25°C)	---	---	---
LiF	(1)	3.65 x 10 <sup>-4</sup> (24.5°C)	---	---	---
MgF <sub>2</sub>	(1)	3.50 x 10 <sup>-4</sup> (24°C)	---	---	---
(n-C <sub>4</sub> H <sub>9</sub> ) <sub>4</sub> NI (Tetra-n-butylammonium iodide)	(5)	1.17 x 10 <sup>-2</sup> (28°C)	Clear	---	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## f. Specific Conductance of Propylene Carbonate Solutions

Solute	Experimental Conditions	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Visual Observations		
			Solution	Solid	Others
Blank		0.8-1.4 x 10 <sup>-5</sup> (*)			
AlCl <sub>3</sub>	(1)	4.52 x 10 <sup>-3</sup> (23°C)	Viscous brown	---	---
	(5)	8.05 x 10 <sup>-3</sup> (33°C)	Brown	---	Exothermic
AlCl <sub>3</sub> +KCl	(4)	8.64 x 10 <sup>-3</sup> (*)	Dark brown	---	---
AlCl <sub>3</sub> +LiCl	(4)	6.61 x 10 <sup>-3</sup> (27°C)	Dark brown	Gray	---
AlCl <sub>3</sub> +MgCl <sub>2</sub>	(4)	6.70 x 10 <sup>-3</sup> (*)	Dark brown	Gray	---
AlCl <sub>3</sub> +CsF	(4)	7.59 x 10 <sup>-3</sup> (*)	Dark brown	Gray	---
AlCl <sub>3</sub> +MgF <sub>2</sub>	(4)	6.68 x 10 <sup>-3</sup> (*)	Dark brown	Gray	---
AlCl <sub>3</sub> +SiF <sub>4</sub>	(7)	6.81 x 10 <sup>-3</sup> (26°C)	Brown	---	Exothermic
AlCl <sub>3</sub> ·NMF	(1)	5.17 x 10 <sup>-5</sup> (24°C)	---	---	---
AlF <sub>3</sub>	(1)	1.17 x 10 <sup>-5</sup> (24°C)	---	---	---
AlF <sub>3</sub> +MgF <sub>2</sub>	(4)	1.27 x 10 <sup>-5</sup> (*)	Clear	Gray	---
AlF <sub>3</sub> +RbF	(4)	2.11 x 10 <sup>-4</sup> (*)	Clear	White	---
AlF <sub>3</sub> ·XH <sub>2</sub> O	(1)	1.24 x 10 <sup>-5</sup> (23°C)	---	---	---
BCl <sub>3</sub> (g)	(2)	1.83 x 10 <sup>-3</sup> (24°C)	Brown	Tan	Exothermic

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## f. Specific Conductance of Propylene Carbonate Solutions (Continued)

Solute	Experimental Conditions	Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Visual Observations		
			Solution	Solid	Others
$\text{BF}_3$ (g)	(2)	$8.68 \times 10^{-4}$ (*)	Light yellow	---	Exothermic
	(6)	$4.43 \times 10^{-4}$ ( $32^\circ\text{C}$ )	Brown	---	---
$\text{BF}_3 + \text{NaF}$	(7)	$6.38 \times 10^{-4}$ ( $27^\circ\text{C}$ )	Brown	Tan	---
$\text{BeCl}_2$	(4)	$4.97 \times 10^{-4}$ (*)	Light green	Light green	Exothermic
$\text{BeCl}_2 + \text{KCl}$	(4)	$4.64 \times 10^{-4}$ (*)	Brown green	Gray	---
$\text{BeCl}_2 + \text{LiCl}$	(4)	$9.85 \times 10^{-4}$ (*)	Cloudy green	White	---
$\text{BeCl}_2 + \text{MgCl}_2$	(4)	$9.72 \times 10^{-4}$ (*)	Dark brown	Gray	---
$\text{BeCl}_2 + \text{CsF}$	(4)	$3.03 \times 10^{-4}$ (*)	Dark brown	Gray	---
$\text{BeCl}_2 + \text{MgF}_2$	(4)	$3.39 \times 10^{-4}$ (*)	Gray	Gray	---
$\text{BeCl}_2 + \text{SiF}_4$	(7)	$5.91 \times 10^{-4}$ ( $25^\circ\text{C}$ )	Light green	Light green	---
$\text{BeF}_2$	(4)	$1.30 \times 10^{-5}$ (*)	Clear	White	---
$\text{BeF}_2 + \text{CsF}$	(4)	$3.59 \times 10^{-4}$ (*)	Clear	Gray	---
$\text{BeF}_2 + \text{KF}$	(4)	$2.59 \times 10^{-5}$ (*)	Clear	White	---
$\text{BeF}_2 + \text{LiF}$	(4)	$1.32 \times 10^{-5}$ (*)	Clear	White	---
$\text{BeF}_2 + \text{MgF}_2$	(4)	$1.34 \times 10^{-5}$ (*)	Clear	White	---
$\text{BeF}_2 + \text{RbF}$	(4)	$2.05 \times 10^{-4}$ (*)	Clear	Gray	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)f. Specific Conductance of Propylene Carbonate Solutions (Continued)

<u>Solute</u>	<u>Experimental Conditions</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )	<u>Solution</u>	<u>Solid</u>	<u>Others</u>
BeF <sub>2</sub> +LiCl	(4)	4.14 x 10 <sup>-4</sup> (*)	Cloudy	White	---
BeF <sub>2</sub> +MgCl <sub>2</sub>	(4)	5.36 x 10 <sup>-4</sup> (*)	Cloudy	Gray	---
CaF <sub>2</sub>	(1)	1.11 x 10 <sup>-5</sup> (24.5°C)	---	---	---
CsCl	(4)	1.20 x 10 <sup>-4</sup> (27°C)	Clear	White	---
CsF	(4)	1.78 x 10 <sup>-4</sup> (*)	Clear	White	---
FeCl <sub>3</sub>	(5)	6.30 x 10 <sup>-3</sup> (*)	Red-brown	Dark brown	---
K <sub>2</sub> BeF <sub>4</sub>	(4)	3.06 x 10 <sup>-5</sup> (27°C)	Clear	White	---
K <sub>2</sub> CO <sub>3</sub>	(1)	5.08 x 10 <sup>-5</sup> (*)	---	---	---
KCl	(4)	2.85 x 10 <sup>-5</sup> (28°C)	Clear	White	---
KF	(4)	2.80 x 10 <sup>-5</sup> (*)	Clear	White	---
KI	(1)	5.25 x 10 <sup>-3</sup> (23.5°C)	Clear	---	---
KPF <sub>6</sub>	(5)	7.24 x 10 <sup>-3</sup> (25°C)	Clear	White	---
KPF <sub>6</sub> +SiF <sub>4</sub>	(7)	7.80 x 10 <sup>-3</sup> (28°C)	Clear	White	---
K <sub>2</sub> SiF <sub>6</sub>	(4)	2.28 x 10 <sup>-5</sup> (*)	Cloudy	White	---
Li <sub>2</sub> CO <sub>3</sub>	(1)	1.22 x 10 <sup>-5</sup> (*)	---	---	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## f. Specific Conductance of Propylene Carbonate Solutions (Continued)

Solute	Experimental Conditions	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Visual Observations		
			Solution	Solid	Others
LiCl	(1)	$3.20 \times 10^{-4}$ (24°C)	---	---	Exothermic
LiF	(1)	$1.23 \times 10^{-5}$ (23°C)	---	---	---
LiPF <sub>6</sub> ·H <sub>2</sub> O	(4)	$1.58 \times 10^{-3}$ (28°C)	Clear	White	---
LiSO <sub>3</sub> F	(4)	$2.47 \times 10^{-4}$ (27°C)	Clear	White	---
Li <sub>2</sub> SiF <sub>6</sub>	(4)	$8.90 \times 10^{-5}$ (27°C)	Clear	White	---
MgCl <sub>2</sub>	(4)	$1.13 \times 10^{-3}$ (*)	Clear	White	---
MgCl <sub>2</sub> ·6H <sub>2</sub> O (dried)	(1)	$2.18 \times 10^{-5}$ (25°C)	---	---	---
MgF <sub>2</sub>	(4)	$1.49 \times 10^{-3}$ (*)	Clear	White	---
MgF <sub>2</sub> ·XH <sub>2</sub> O	(1)	$7.65 \times 10^{-6}$ (24.5°C)	Milky	---	---
MgSiF <sub>6</sub>	(4)	$8.93 \times 10^{-5}$ (*)	Clear	White	---
Na <sub>3</sub> AlF <sub>6</sub>	(4)	$1.02 \times 10^{-5}$ (27°C)	Clear	White	---
NaBF <sub>4</sub>	(4)	$1.77 \times 10^{-3}$ (27°C)	Clear	White	---
NaCl	(4)	$1.69 \times 10^{-5}$ (28°C)	Clear	White	---
NaF	(4)	$1.15 \times 10^{-5}$ (*)	Cloudy	White	---
NaPF <sub>6</sub>	(4)	$7.53 \times 10^{-3}$ (29°C)	Clear	Tan	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## f. Specific Conductance of Propylene Carbonate Solutions (Continued)

Solute	Experimental Conditions	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Visual Observations		
			Solution	Solid	Others
Na <sub>2</sub> SiF <sub>6</sub>	(4)	1.23 x 10 <sup>-5</sup> (*)	Clear	White	---
RbF	(4)	1.05 x 10 <sup>-4</sup> (*)	Clear	White	---
SiF <sub>4</sub> (g)	(6)	4.50 x 10 <sup>-4</sup> (27°C)	Light yellow	---	Exothermic
SiF <sub>4</sub> +AlCl <sub>3</sub>	(7)	6.52 x 10 <sup>-3</sup> (26°C)	Brown	---	Exothermic
SiF <sub>4</sub> +BeCl <sub>2</sub>	(7)	5.48 x 10 <sup>-4</sup> (26°C)	Light green	Green	Exothermic
SiF <sub>4</sub> +RbF	(7)	1.27 x 10 <sup>-4</sup> (28°C)	Clear	White	---
SiF <sub>4</sub> +TiF <sub>4</sub>	(7)	3.38 x 10 <sup>-3</sup> (28°C)	Tan	Tan	---
TiF <sub>3</sub>	(4)	3.67 x 10 <sup>-4</sup> (*)	Brown	Dark brown	---
TiF <sub>4</sub>	(4)	3.50 x 10 <sup>-3</sup> (*)	Brown	Tan	---
(n-C <sub>4</sub> H <sub>9</sub> ) <sub>4</sub> NI (Tetra-n-butylammonium iodide)	(5)	5.23 x 10 <sup>-3</sup> (*)	Clear yellow	---	---

(\*) Room Temperature



TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## g. Specific Conductance of Tetramethylurea Solutions

Solute	Experimental Conditions	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Visual Observations		
			Solution	Solid	Others
Blank		$2 \times 10^{-6}$ (*)			
AlCl <sub>3</sub>	(4)	$8.28 \times 10^{-3}$ (48°C)	Tan	---	Solidified overnight
CsPF <sub>6</sub>	(4)	$5.90 \times 10^{-3}$ (29°C)	Clear	---	---
FeCl <sub>3</sub>	(4)	$2.40 \times 10^{-3}$ (29°C)	Brown	Brown	---
KAsF <sub>6</sub>	(5)	$6.59 \times 10^{-3}$ (30°C)	Milky	White	---
KPF <sub>6</sub>	(5)	$7.40 \times 10^{-3}$ (29°C)	Clear	---	Exothermic
LiCl	(4)	$6.48 \times 10^{-3}$ (28°C)	Clear	White	---
LiPF <sub>6</sub> ·H <sub>2</sub> O	(4)	$3.33 \times 10^{-4}$ (28°C)	Clear	White	---
LiSO <sub>3</sub> F	(4)	$3.49 \times 10^{-4}$ (28°C)	Clear	Gray	---
MgCl <sub>2</sub>	(4)	$4.01 \times 10^{-4}$ (28°C)	Clear	White	---
MgSiF <sub>6</sub>	(4)	$2.18 \times 10^{-3}$ (28°C)	Clear	White	---
NH <sub>4</sub> SO <sub>3</sub> F	(4)	$5.10 \times 10^{-3}$ (30°C)	Clear	White	---
NaBF <sub>4</sub>	(4)	$4.83 \times 10^{-3}$ (28°C)	Clear	White	---
NaPF <sub>6</sub>	(4)	$6.83 \times 10^{-3}$ (28°C)	Tan	Tan	Exothermic
NaSbF <sub>6</sub>	(5)	$6.75 \times 10^{-3}$ (30°C)	Milky	White	---

(\*) Room Temperature

TABLE IV. SPECIFIC CONDUCTANCE OF NONAQUEOUS SOLUTIONS (Continued)

## g. Specific Conductance of Tetramethylurea Solutions (Continued)

Solute	Experimental Conditions	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Visual Observations		
			Solution	Solid	Others
$\begin{array}{c} \text{CH}_2\text{CH}_2 \diagup \text{NH}_2 \\ \text{O} \diagdown \text{CH}_2\text{CH}_2 \end{array} \text{PF}_6$ (Morpholinium hexafluorophosphate)	(5)	$8.93 \times 10^{-3}$ (29°C)	Amber	---	---
$(\text{n-C}_4\text{H}_9)_4\text{NI}$ (Tetra-n-butylammonium iodide)	(4)	$1.78 \times 10^{-3}$ (27.5°C)	Clear	Cream	---

TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS

	<u>Page No.</u>
a. $\text{NaPF}_6$ in Dimethyl Cyanamide . . . . .	III-36
b. Di-n-butylammonium Hexafluoroarsenate in Dimethyl Cyanamide . . . . .	III-37
c. N-benzyl N,N,N, - Trimethylammonium Hexafluoro- antimonate in Dimethyl Cyanamide . . . . .	III-38
d. $\text{CsPF}_6$ in Dimethylformamide . . . . .	III-39
e. $\text{LiCl}$ in Dimethylformamide . . . . .	III-40
f. $\text{KPF}_6$ in Dimethylformamide . . . . .	III-41
g. $\text{NaBF}_4$ in Dimethylformamide . . . . .	III-42
h. $\text{NaPF}_6$ in Dimethylformamide . . . . .	III-43
i. $\text{SbCl}_3$ in Dimethylformamide . . . . .	III-44
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k. $\text{KPF}_6$ in Propylene Carbonate . . . . .	III-46
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All tests were performed in a dry argon atmosphere unless otherwise specified.

TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS

a. NaPF<sub>6</sub>-Dimethyl Cyanamide

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )	<u>Viscosity (25°C)</u> (Centipoises)
1.00	1.96 x 10 <sup>-2</sup> (27°C)	---
1.25	2.08 x 10 <sup>-2</sup> (28°C)	---
1.48	2.12 x 10 <sup>-2</sup> (29°C)	---
1.70	2.12 x 10 <sup>-2</sup> (29°C)	1.97
2.04	2.04 x 10 <sup>-2</sup> (28°C)	---
2.38	1.94 x 10 <sup>-2</sup> (28.5°C)	2.99

Note: Solid residue present in every sample.

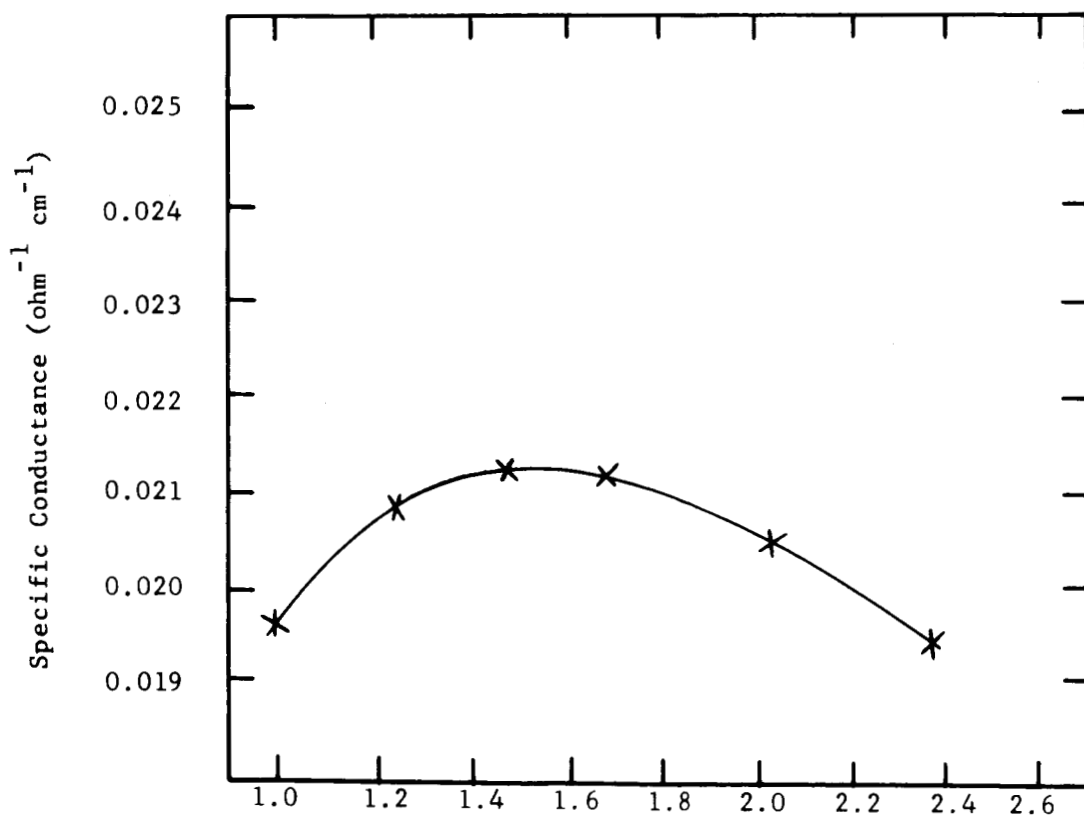


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

b. Di-n-butylammonium Hexafluoroarsenate-Dimethyl Cyanamide

<u>Molality</u>	<u>Specific Conductance</u> <u>(ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Viscosity (25°C)</u> <u>(Centipoises)</u>
1.00	$2.31 \times 10^{-2}$ (28°C)	---
1.24	$2.43 \times 10^{-2}$ (28°C)	---
1.48	$2.50 \times 10^{-2}$ (28.5°C)	---
1.71	$2.51 \times 10^{-2}$ (28°C)	---
1.95	$2.49 \times 10^{-2}$ (28°C)	---
2.31	$2.42 \times 10^{-2}$ (28.5°C)	2.28

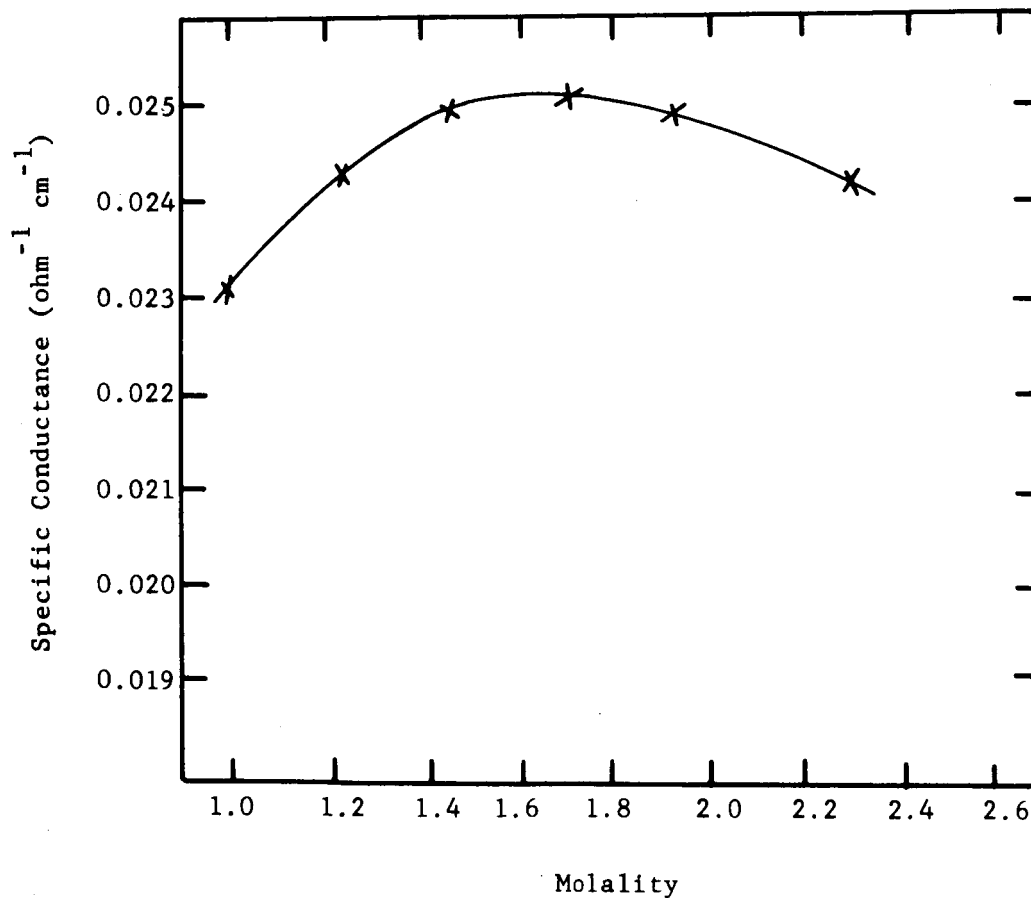


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

c. N-benzyl N,N,N-trimethylammonium Hexafluoroantimonate-Dimethyl Cyanamide

<u>Molality</u>	<u>Specific Conductance</u> ( $\text{ohm}^{-1} \text{cm}^{-1}$ )	<u>Viscosity (25°C)</u> (Centipoises)
1.00	$1.92 \times 10^{-2}$ (28°C)	---
1.20	$1.98 \times 10^{-2}$ (28°C)	---
1.40	$2.00 \times 10^{-2}$ (28°C)	---
1.61	$1.99 \times 10^{-2}$ (28°C)	1.91
1.90	$1.95 \times 10^{-2}$ (28°C)	---
2.20	$1.91 \times 10^{-2}$ (29°C)	2.48

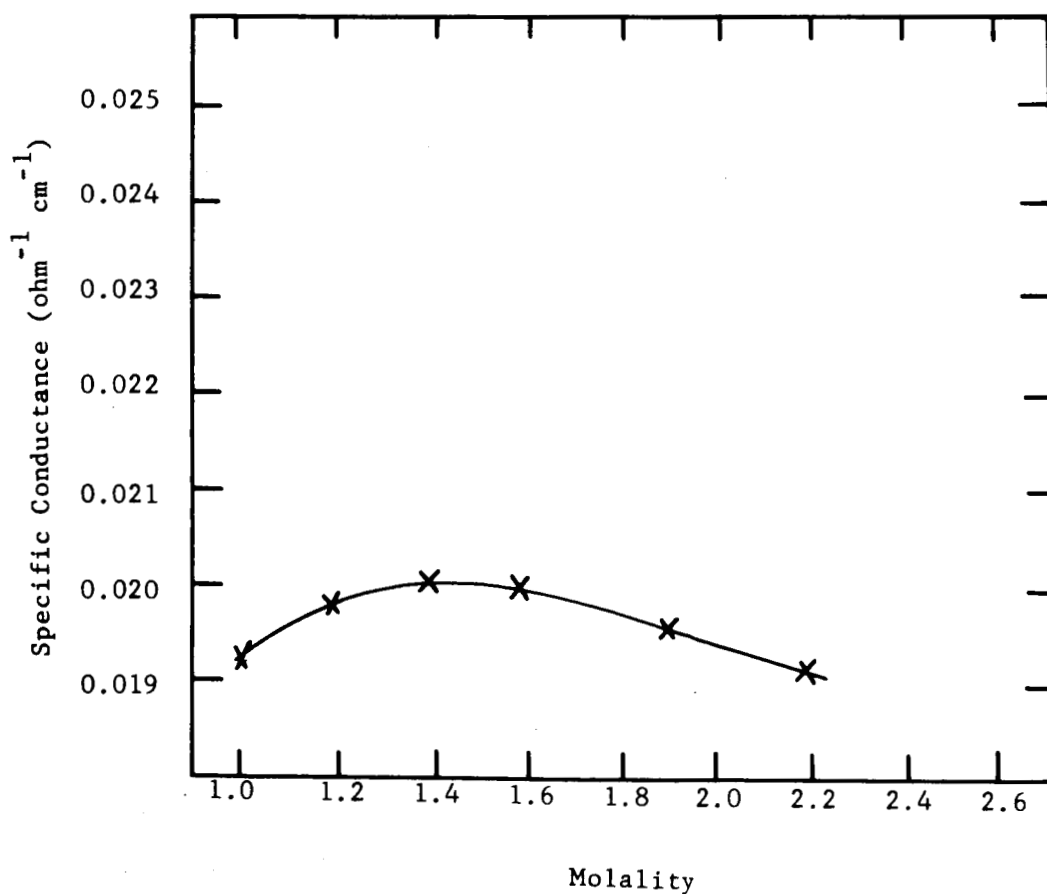


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

d. CsPF<sub>6</sub> in Dimethylformamide

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )
0.382	1.44 x 10 <sup>-2</sup> (30°C)
0.764	2.27 x 10 <sup>-2</sup> (30°C)
1.14	2.61 x 10 <sup>-2</sup> (30°C)
1.375	2.55 x 10 <sup>-2</sup> (28°C)
1.53	2.61 x 10 <sup>-2</sup> (30°C)
1.715 (Solid Present)	2.46 x 10 <sup>-2</sup> (28°C)
2.29 (Solid Present)	2.54 x 10 <sup>-2</sup> (30°C)

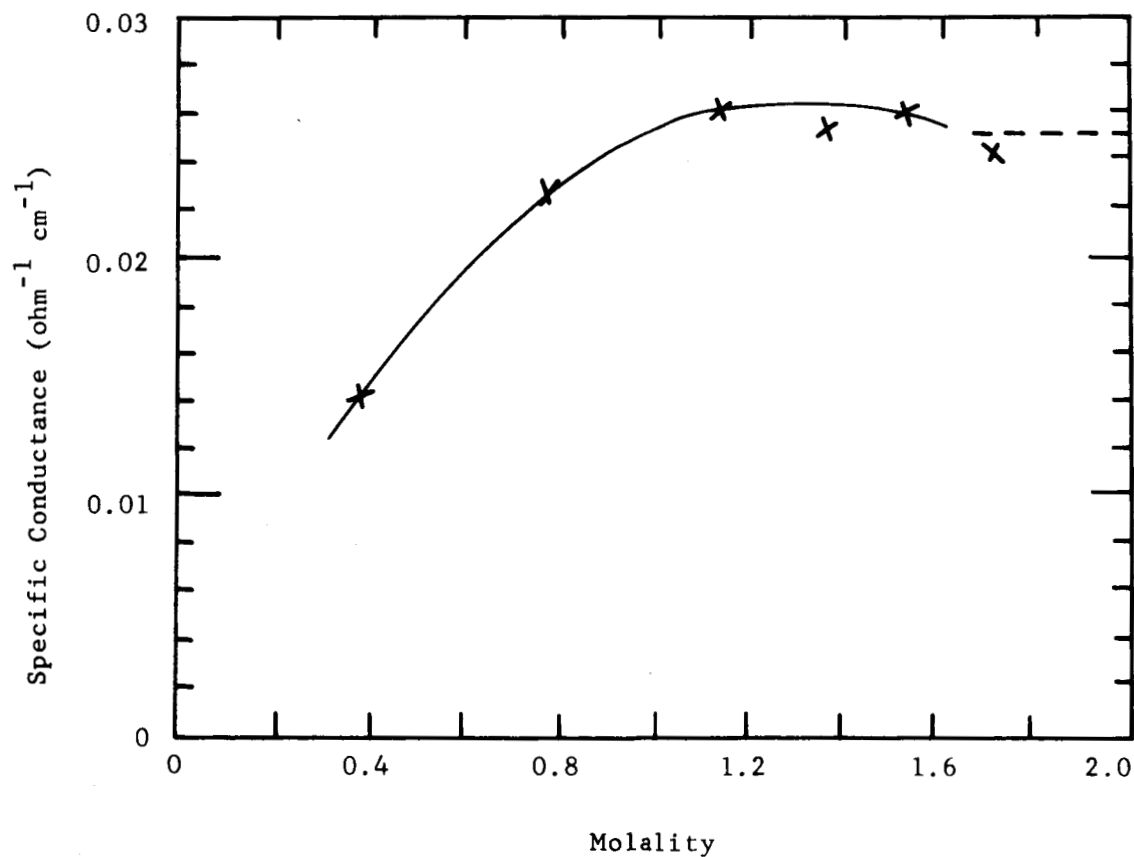


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

e. LiCl in Dimethylformamide

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )
0.0176	0.756 x 10 <sup>-3</sup>
0.1057	3.12 x 10 <sup>-3</sup>
0.526	7.10 x 10 <sup>-3</sup>
0.672	7.66 x 10 <sup>-3</sup>
1.052	8.01 x 10 <sup>-3</sup>
2.186	5.09 x 10 <sup>-3</sup>
2.667	3.59 x 10 <sup>-3</sup>
3.035	3.22 x 10 <sup>-3</sup>

Note: This test was performed in open atmosphere at room temperature.

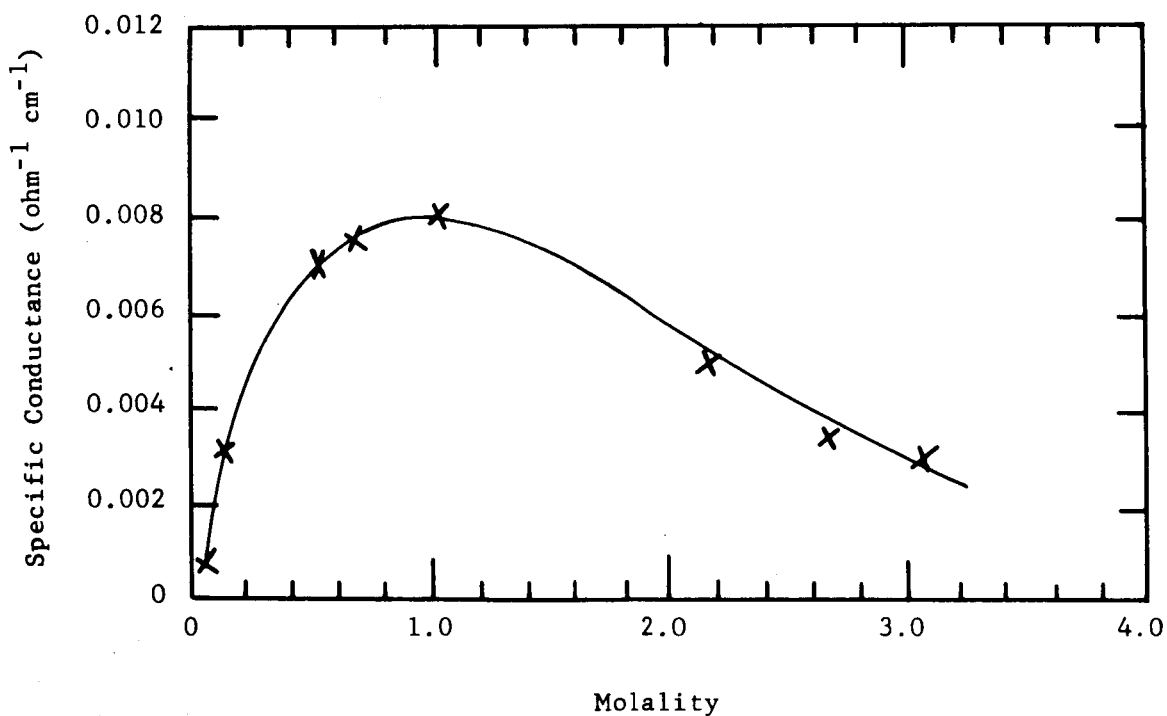




TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

f.  $\text{KPF}_6$  in Dimethylformamide

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )	<u>Viscosity (25°C)</u> (Centipoises)
0.0109	$0.0714 \times 10^{-2}$ (27°C)	0.840
0.0963	$0.506 \times 10^{-2}$ (27°C)	0.895
0.498	$1.73 \times 10^{-2}$ (27°C)	1.206
0.76	$2.15 \times 10^{-2}$ (27°C)	1.42
0.99	$2.36 \times 10^{-2}$ (27°C)	1.68
1.25	$2.47 \times 10^{-2}$ (27°C)	2.02
1.50	$2.48 \times 10^{-2}$ (27°C)	2.51
1.76	$2.44 \times 10^{-2}$ (27°C)	2.92
2.19	$2.31 \times 10^{-2}$ (27°C)	3.52
2.97	$1.80 \times 10^{-2}$ (29°C)	7.18
3.99	$1.20 \times 10^{-2}$ (28°C)	14.7

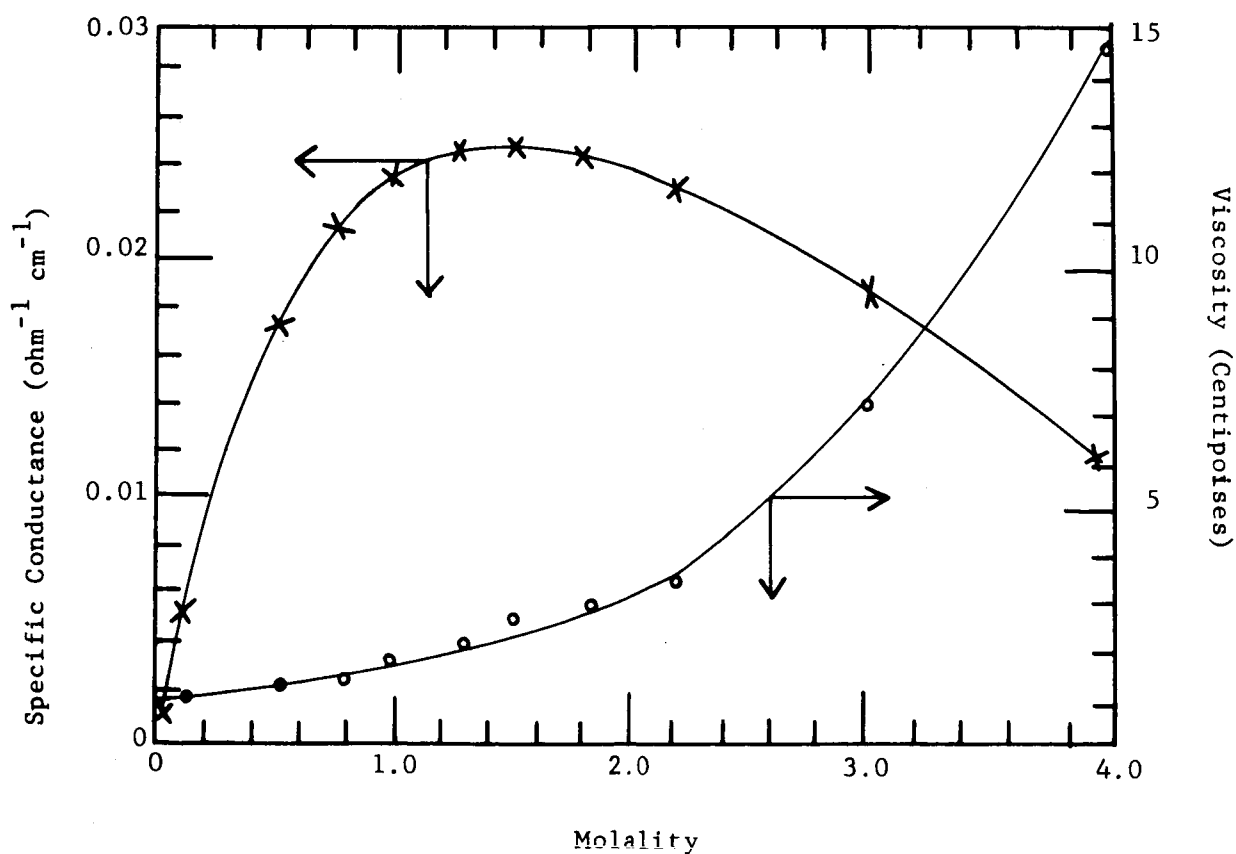


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

g. NaBF<sub>4</sub> in Dimethylformamide

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )	<u>Viscosity (25°C)</u> (Centipoises)
0.0196	0.109 x 10 <sup>-2</sup> (28°C)	0.923
0.174	0.715 x 10 <sup>-2</sup> (29°C)	
0.610	1.63 x 10 <sup>-2</sup> (26°C)	1.67
1.01	2.04 x 10 <sup>-2</sup> (27°C)	2.10
1.48	2.27 x 10 <sup>-2</sup> (28°C)	3.03
1.92	2.17 x 10 <sup>-2</sup> (28°C)	3.47
3.02	1.81 x 10 <sup>-2</sup> (28°C)	4.67
4.12	1.33 x 10 <sup>-2</sup> (28°C)	9.97

Note: Solid present in every sample (8-11%).

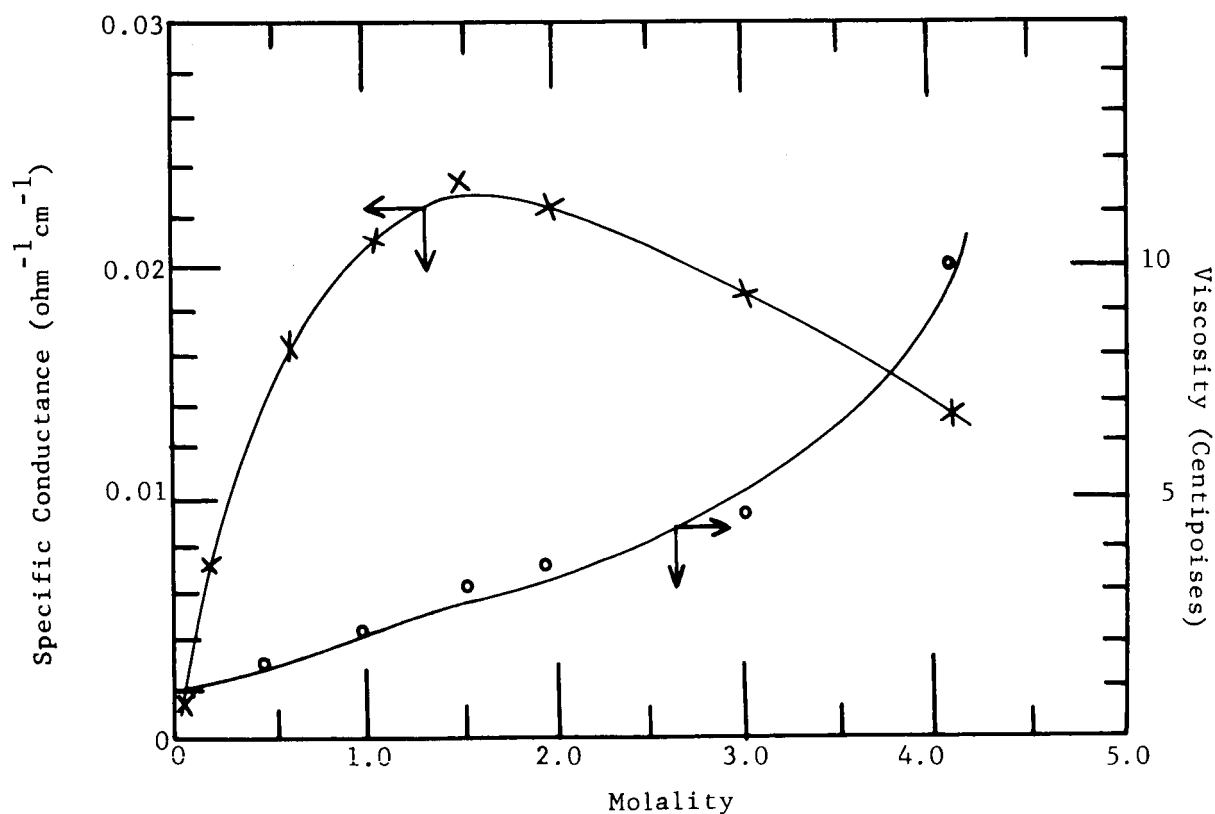


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

h. NaPF<sub>6</sub> in Dimethylformamide

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )
0.401	1.42 x 10 <sup>-2</sup> (27°C)
0.995	2.33 x 10 <sup>-2</sup> (29°C)
* 1.00	2.14 x 10 <sup>-2</sup> (29°C)
1.496	2.31 x 10 <sup>-2</sup> (27°C)
* 1.50	2.24 x 10 <sup>-2</sup> (29°C)
1.98	2.36 x 10 <sup>-2</sup> (29°C)
* 2.00	2.10 x 10 <sup>-2</sup> (29°C)
2.36	2.00 x 10 <sup>-2</sup> (27°C)
2.88	1.59 x 10 <sup>-2</sup> (27°C)
4.02	0.475 x 10 <sup>-2</sup> (29°C)

\* Different batch of NaPF<sub>6</sub> used.

NOTE: Solid present in every sample (8-11%).

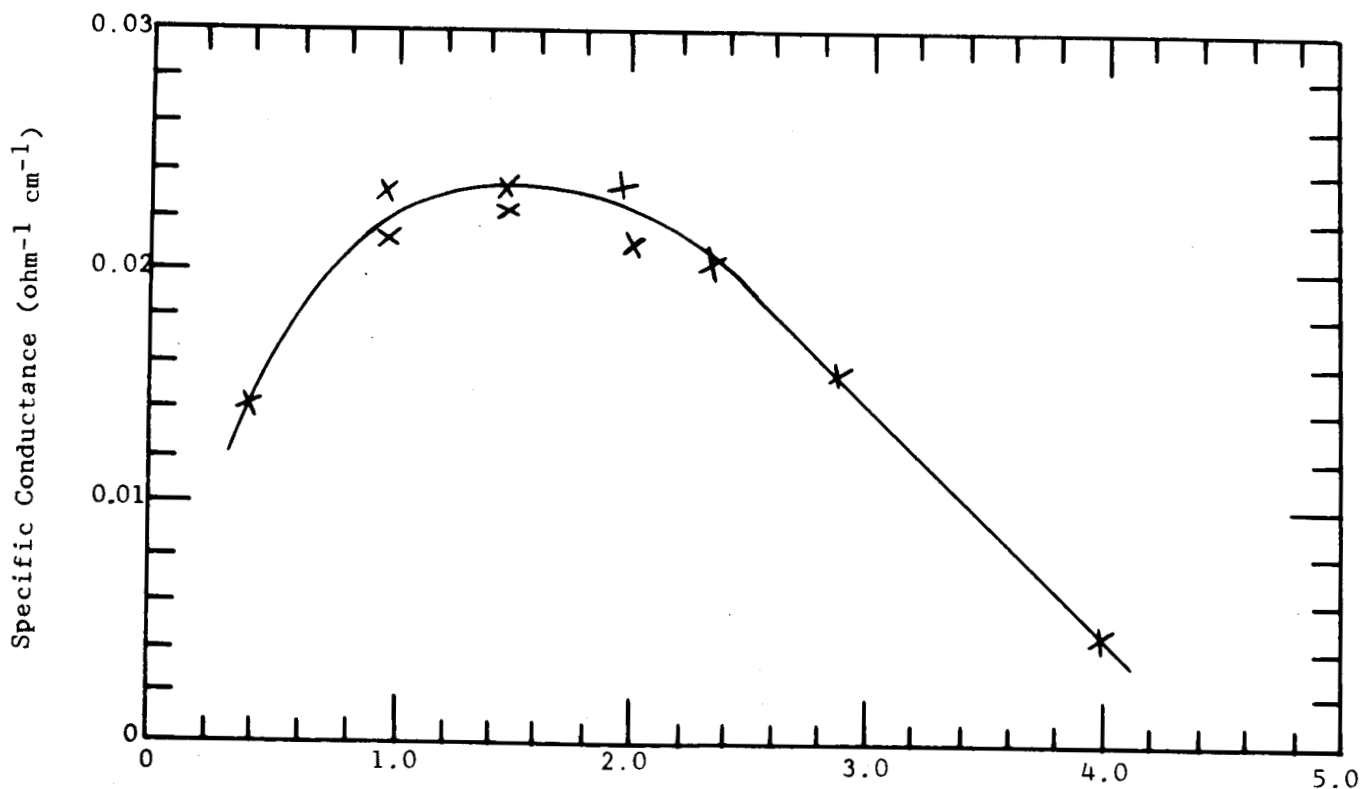


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

i. SbCl<sub>3</sub> in Dimethylformamide

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )
1.00	1.50 x 10 <sup>-3</sup> (26°C)
1.20	1.59 x 10 <sup>-3</sup> (26°C)
1.40	1.65 x 10 <sup>-3</sup> (26°C)
1.60	1.68 x 10 <sup>-3</sup> (26°C)
1.80	1.68 x 10 <sup>-3</sup> (26°C)
2.00	1.66 x 10 <sup>-3</sup> (26°C)
2.20	1.61 x 10 <sup>-3</sup> (26°C)

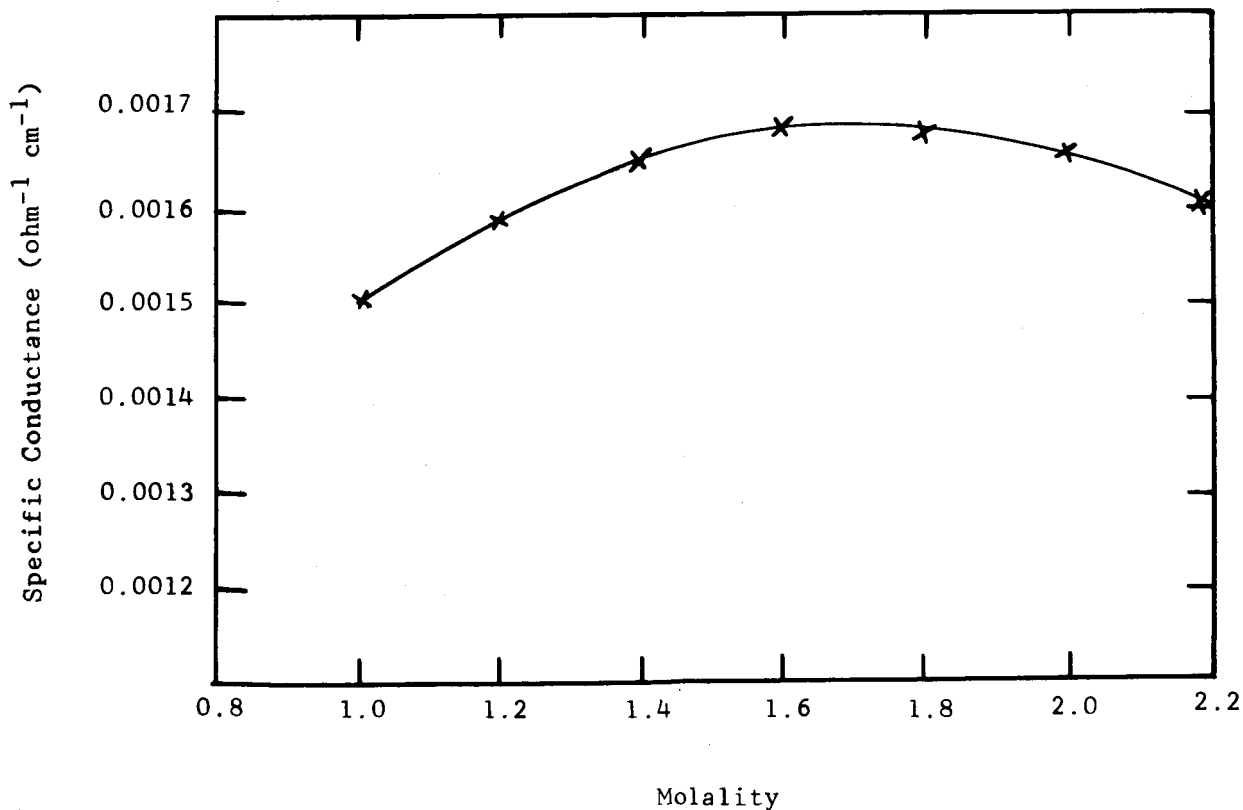


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

j. Morpholinium Hexafluorophosphate in Dimethylformamide

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )
1.0	2.57 x 10 <sup>-2</sup> (27°C)
2.0	2.91 x 10 <sup>-2</sup> (28°C)
3.0	2.67 x 10 <sup>-2</sup> (29°C)
4.0	2.23 x 10 <sup>-2</sup> (30°C)
5.0	1.66 x 10 <sup>-2</sup> (28.5°C)
6.0	1.29 x 10 <sup>-2</sup> (30°C)

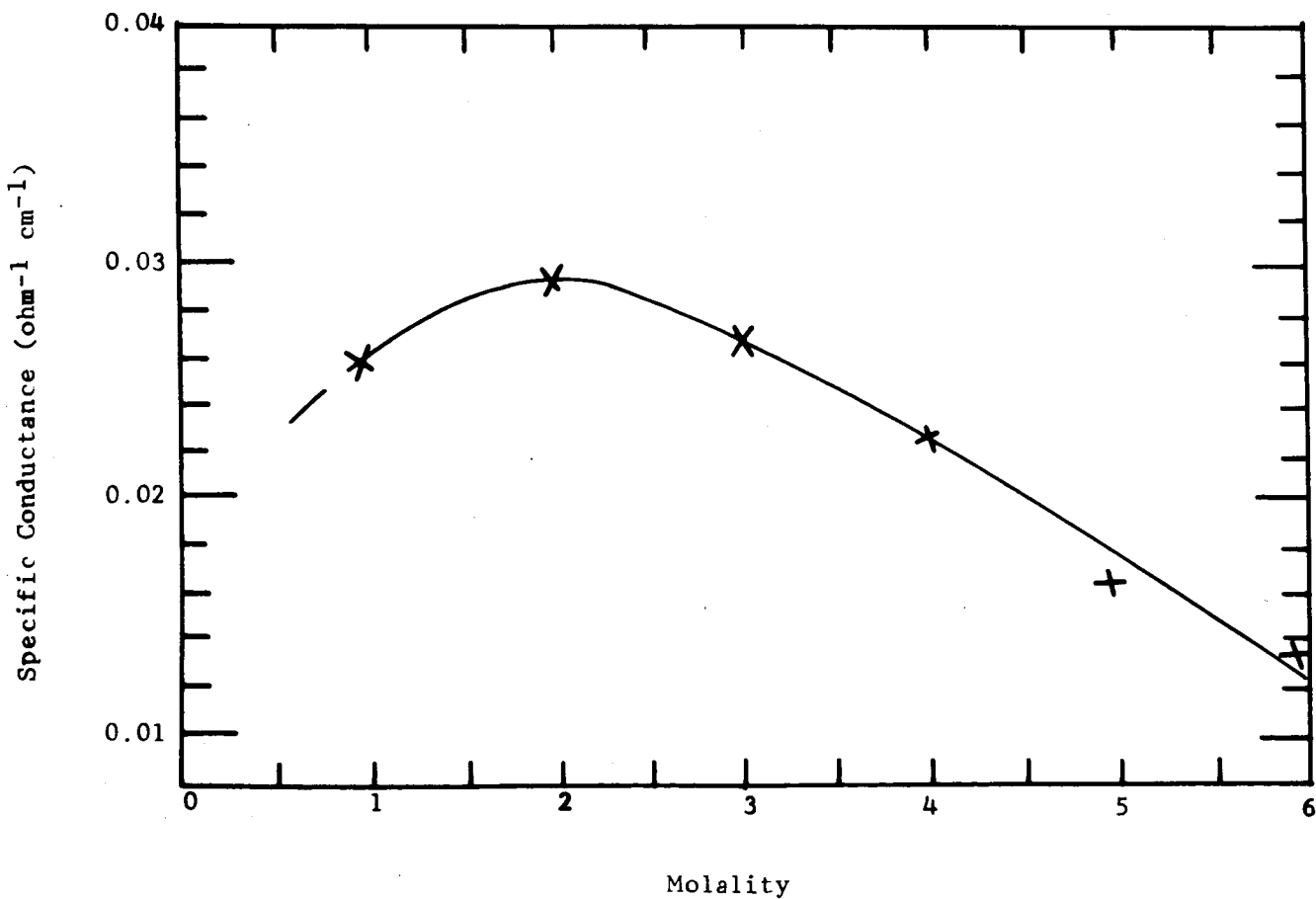


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

k. KPF<sub>6</sub> in Propylene Carbonate

<u>Molality</u>	<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )	<u>Viscosity (25°C)</u> (Centipoises)
0.0	---	2.52
0.216	4.11 x 10 <sup>-3</sup> (25°C)	3.06
0.40	5.78 x 10 <sup>-3</sup> (25°C)	3.66
0.603	6.78 x 10 <sup>-3</sup> (25°C)	4.38
0.80	7.19 x 10 <sup>-3</sup> (25°C)	5.10
1.00	7.24 x 10 <sup>-3</sup> (25°C)	5.88
1.20	7.30 x 10 <sup>-3</sup> (25°C) (Saturated)	---
1.40	7.29 x 10 <sup>-3</sup> (25°C) (Saturated)	6.60

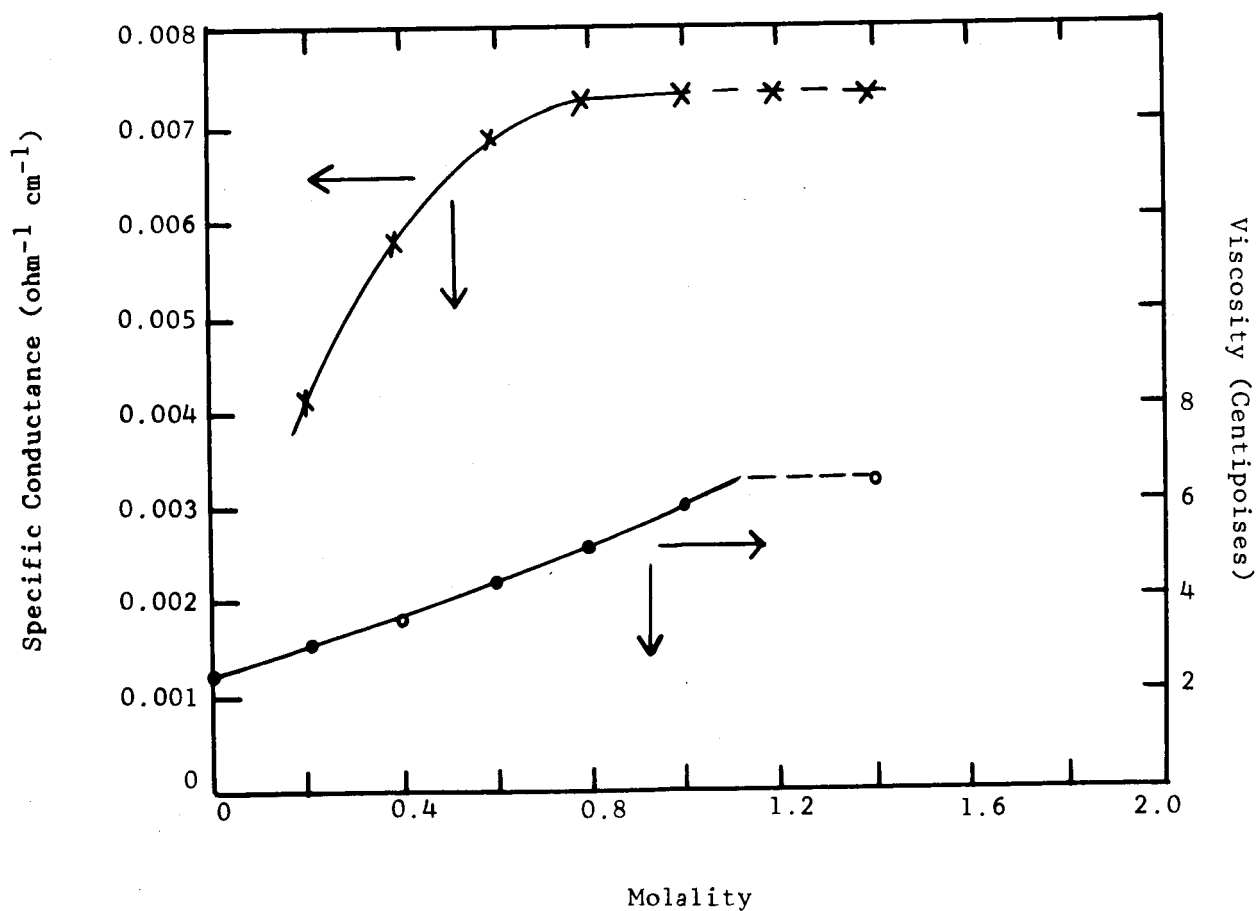
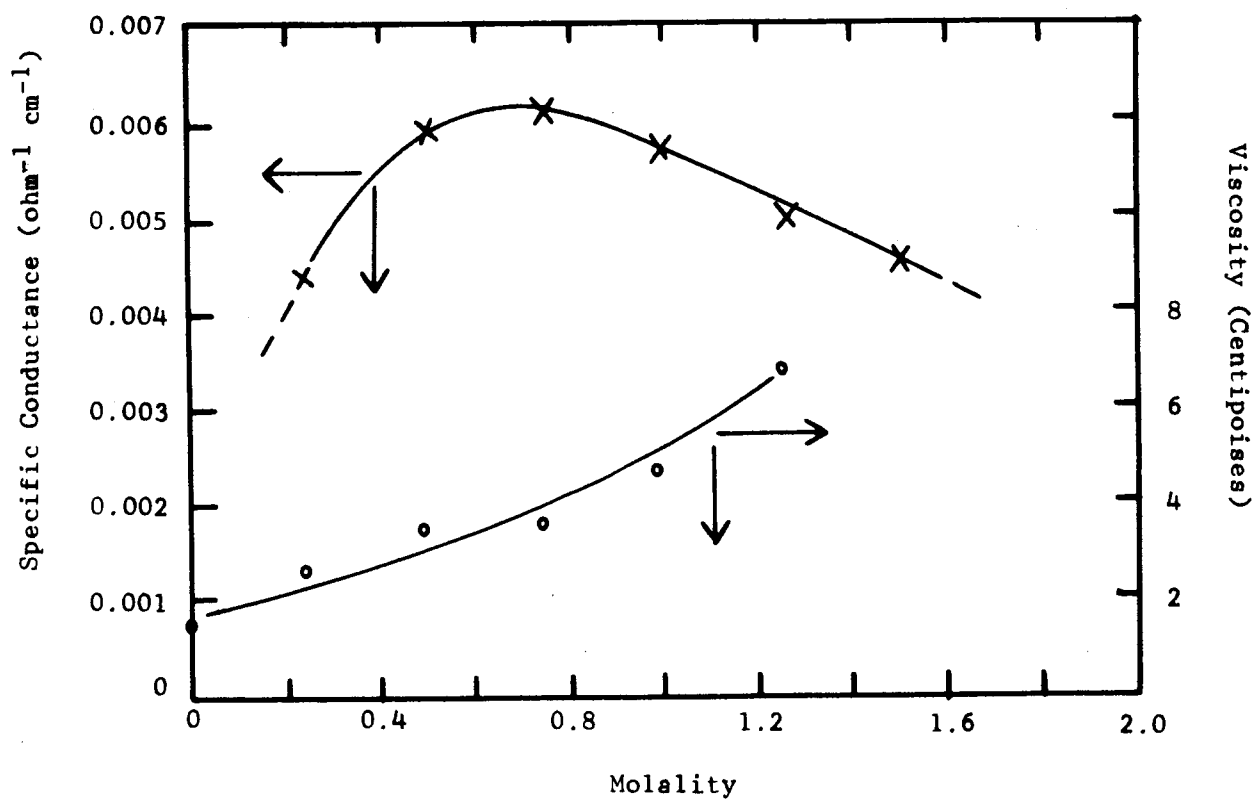


TABLE V. SPECIFIC CONDUCTANCE AND VISCOSITY  
VS. CONCENTRATION OF SOLUTIONS (Continued)

1.  $KPF_6$  in Tetramethylurea

<u>Molality</u>	<u>Specific Conductance (28°C)</u> <u>(ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Viscosity (25°C)</u> <u>(Centipoises)</u>
0.0	---	1.41
0.248	$4.38 \times 10^{-3}$	2.61
0.499	$5.89 \times 10^{-3}$	3.47
0.746	$6.06 \times 10^{-3}$	3.50
1.0	$5.68 \times 10^{-3}$	4.82
1.26	$5.00 \times 10^{-3}$	6.80
1.50	$4.07 \times 10^{-3}$	---



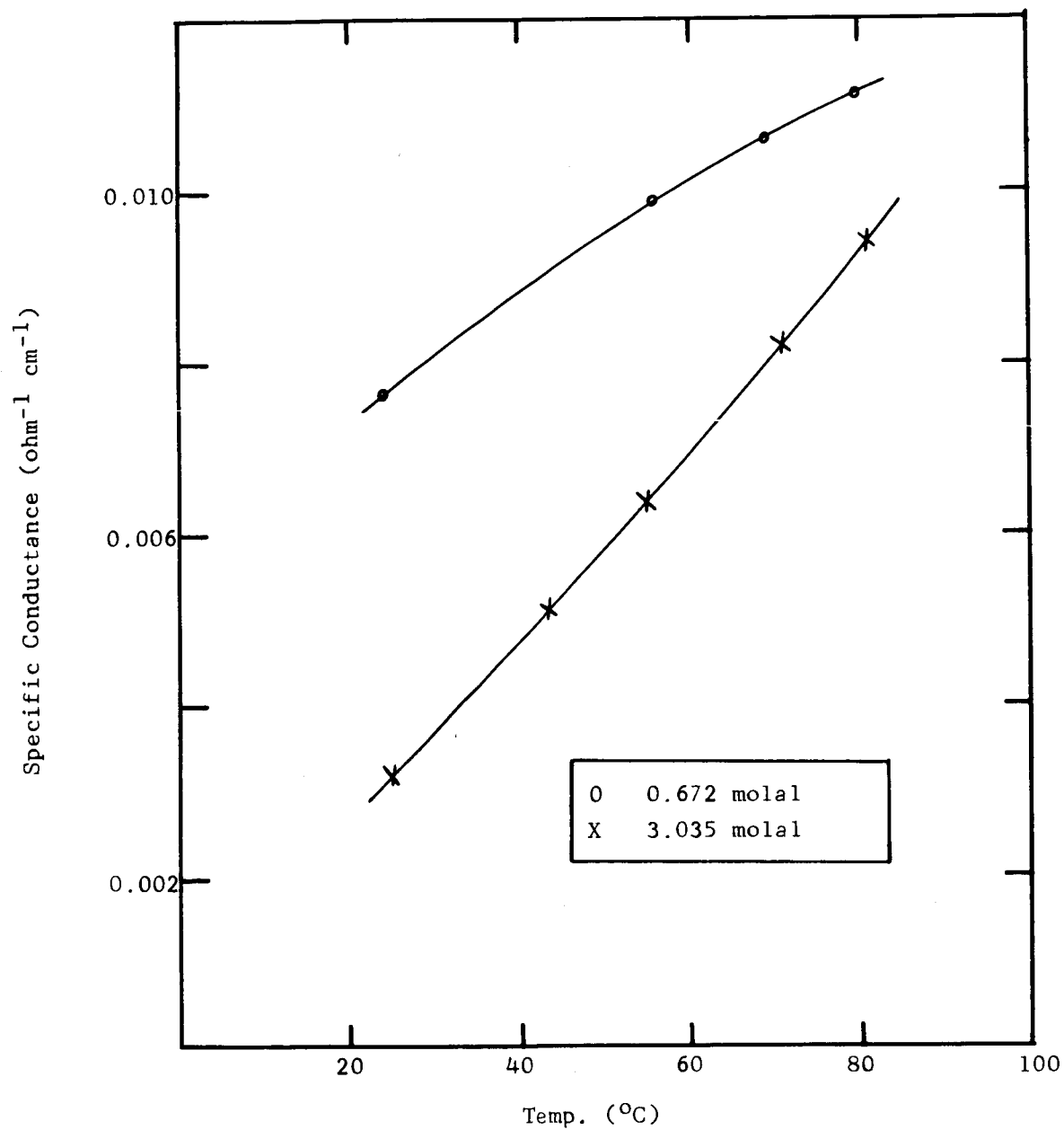


FIGURE 8. TEMPERATURE COEFFICIENTS OF CONDUCTANCE  
OF LiCl-DIMETHYLFORMAMIDE SOLUTIONS



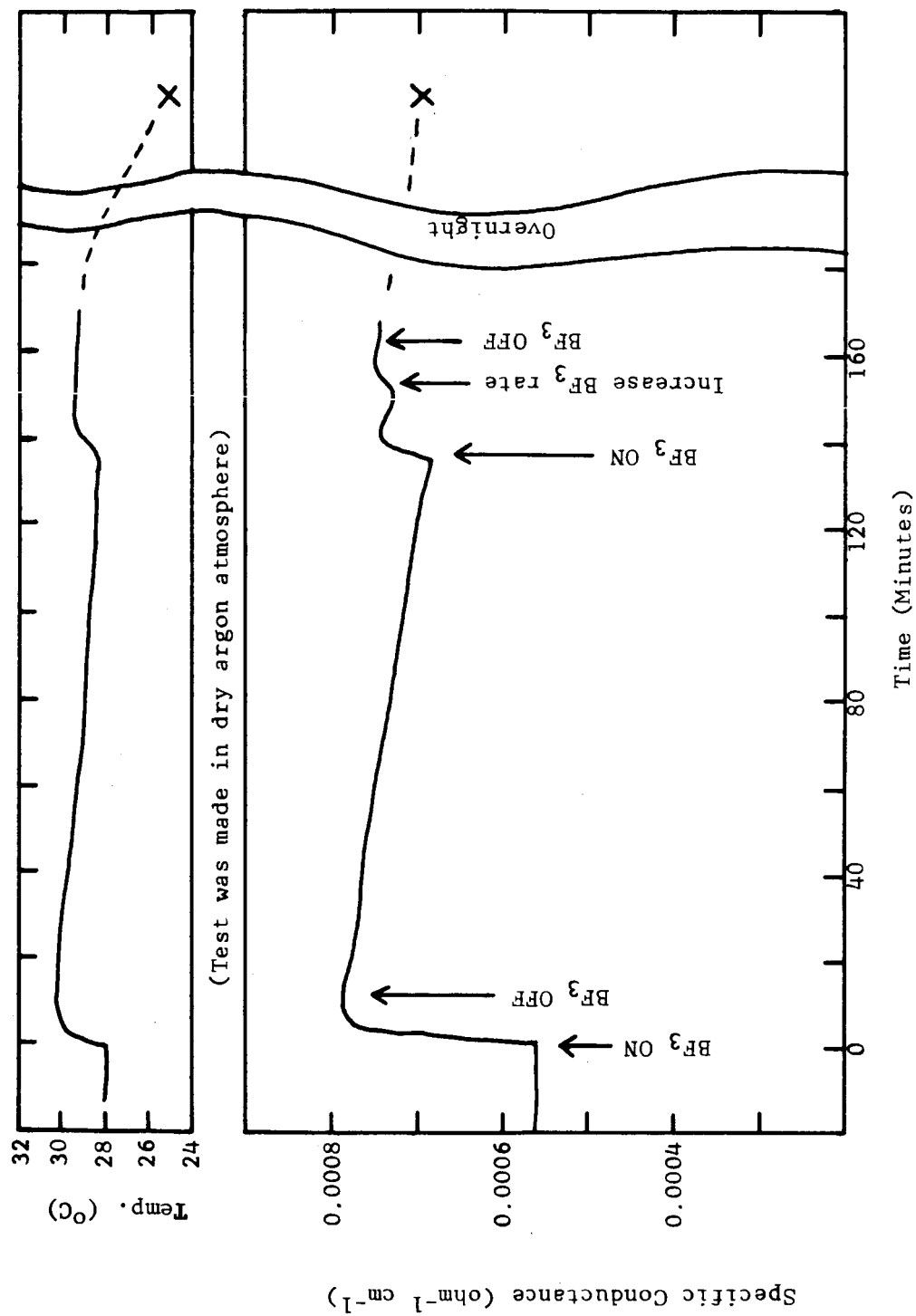


FIGURE 9. EFFECT OF EXCESS BF<sub>3</sub> ON CONDUCTANCE OF 47% BF<sub>3</sub>-ETHER COMPLEX

TABLE VI. FORMATION OF ETHER  
COMPLEXES OF SOLUTES

M-DIMETHOXYBENZENE

<u>Solute</u>	<u>Specific Conductance</u> <u>(ohm<sup>-1</sup> cm<sup>-1</sup>)</u>		<u>Observations</u>
	<u>1 Hr. Exposure</u>		
Blank 1	1.94 x 10 <sup>-9</sup> (R.T.)		---
Blank 2	2.53 x 10 <sup>-9</sup> (R.T.)		---
BeCl <sub>2</sub>	1.33 x 10 <sup>-6</sup> (31°C)		Yellow, exothermic reaction
BeF <sub>2</sub>	2.51 x 10 <sup>-9</sup> (27°C)		No apparent reaction
BCl <sub>3</sub>	3.24 x 10 <sup>-4</sup> (24.5°C)		Red, exothermic reaction
BF <sub>3</sub>	3.32 x 10 <sup>-5</sup> (42.5°C)		Orange color, exothermic reaction

BIS [2-(2-METHOXYETHOXY)ETHYL] ETHER

	<u>1 Hr. Exposure</u>	<u>24 Hr. Exposure</u>	
Blank 1	4.60 x 10 <sup>-9</sup> (R.T.)	1.62 x 10 <sup>-9</sup> (18°C)	---
Blank 2	1.21 x 10 <sup>-9</sup> (19.5°C)	---	---
BeCl <sub>2</sub>	1.49 x 10 <sup>-5</sup> (38°C)	1.66 x 10 <sup>-9</sup> (18°C)	Brown, exothermic reaction
BeF <sub>2</sub>	1.77 x 10 <sup>-7</sup> (30°C)	1.27 x 10 <sup>-9</sup> (27°C)	Milky, slightly exothermic reaction
BCl <sub>3</sub>	2.64 x 10 <sup>-9</sup> (54°C)	---	Dark brown, exothermic reaction
BF <sub>3</sub>	1.91 x 10 <sup>-9</sup> (71°C)	---	Brown, exothermic reaction

TABLE VII. MIXED SOLVENT TEST RESULTS

	<u>Page No.</u>
a. $\text{KPF}_6$ -DMF-Acetone . . . . .	III-52
b. $\text{NaPF}_6$ -DMF-Acetone . . . . .	III-53

All tests were performed in a dry argon atmosphere.

TABLE VII. MIXED SOLVENT TEST RESULTSa. KPF<sub>6</sub>-DMF-Acetone

Solvent		Specific Conductance (28.5°C) (ohm <sup>-1</sup> cm <sup>-1</sup> )
Weight % DMF	Weight % Acetone	
0.0	100	$2.79 \times 10^{-2}$
18.9	81.1	$2.95 \times 10^{-2}$
36.9	63.1	$3.06 \times 10^{-2}$
53.8	46.2	$2.94 \times 10^{-2}$
70.0	30.0	$2.90 \times 10^{-2}$
85.3	14.7	$2.70 \times 10^{-2}$
100	0.0	$2.47 \times 10^{-2}$

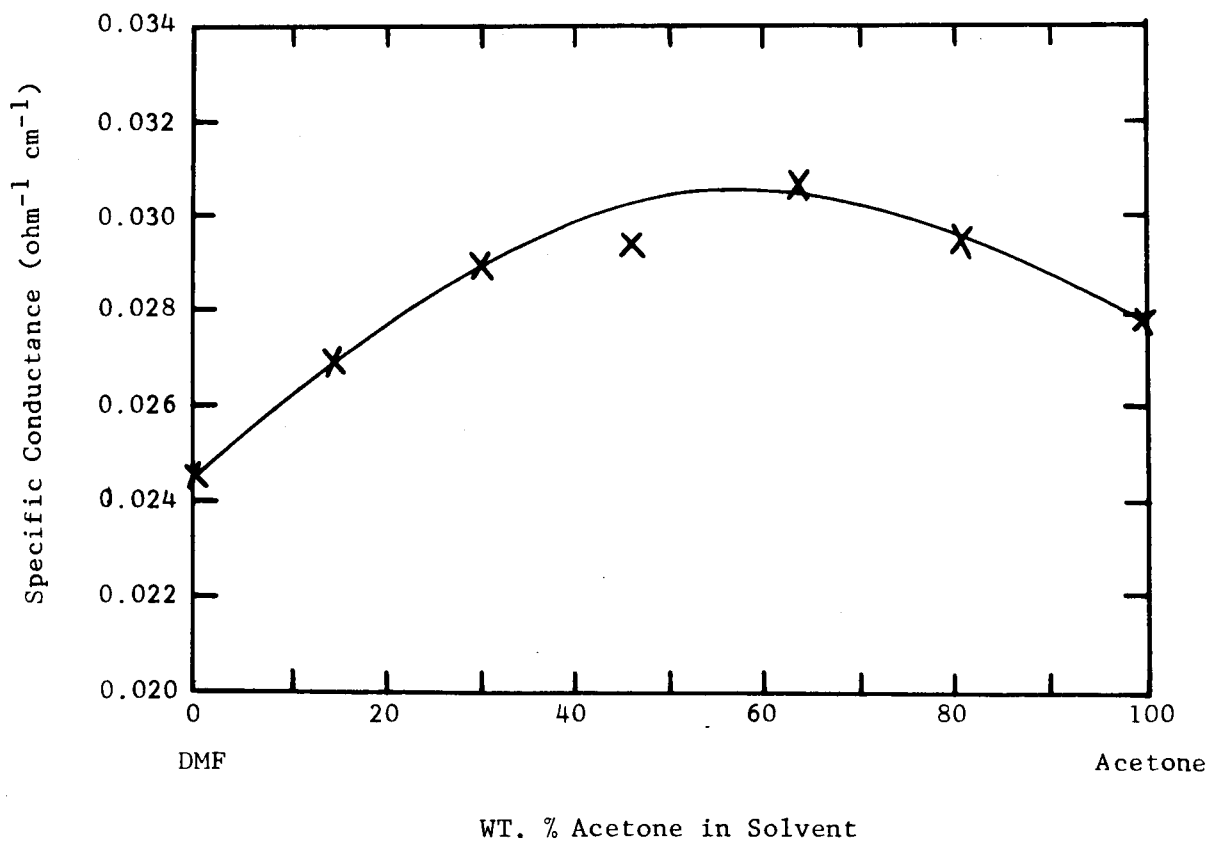
Concentration of KPF<sub>6</sub> = 1.5 molal.

TABLE VII. MIXED SOLVENT TEST RESULTS (Continued)b. NaPF<sub>6</sub>-DMF-Acetone

Solvent		Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )
Weight % DMF	Weight % Acetone	
0.0	100	$3.54 \times 10^{-2}$ (28.5°C)
18.5	81.5	$3.50 \times 10^{-2}$ (28°C)
36.2	63.8	$3.18 \times 10^{-2}$ (28°C)
53.2	46.8	$3.01 \times 10^{-2}$ (28°C)
69.8	30.2	$2.59 \times 10^{-2}$ (28°C)
85.3	14.7	$2.39 \times 10^{-2}$ (28°C)
100	0.0	$2.16 \times 10^{-2}$ (29°C)

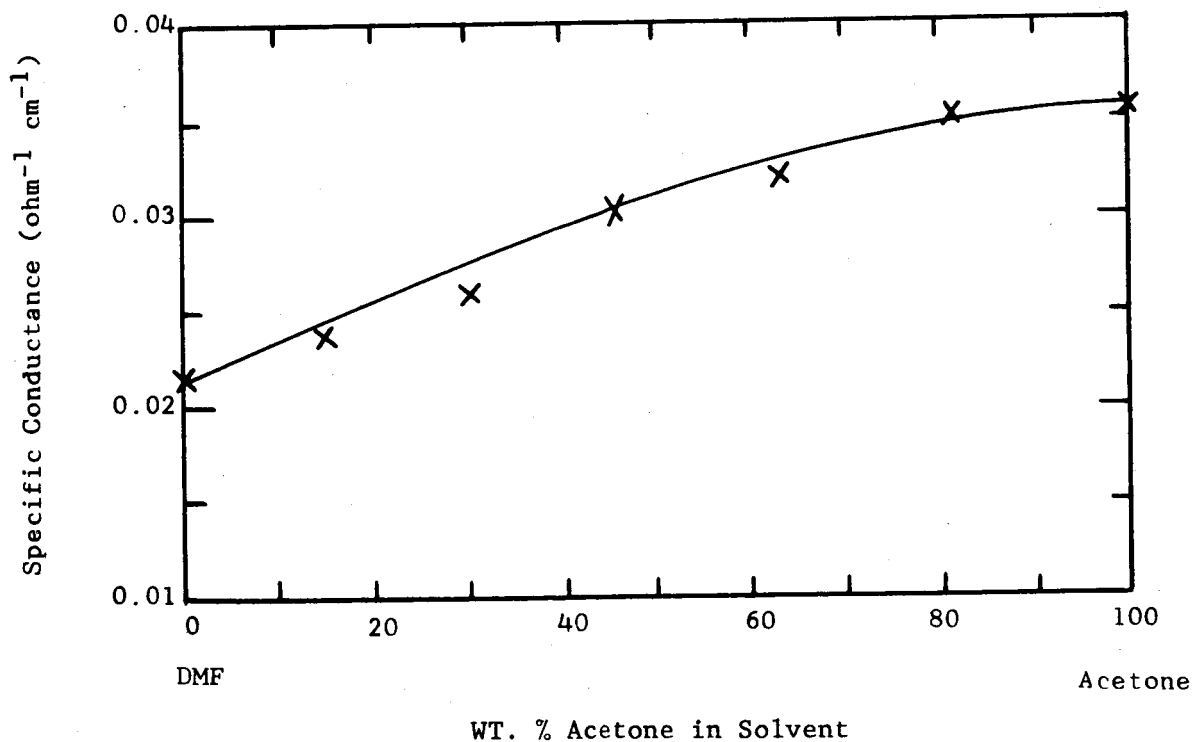
Concentration of NaPF<sub>6</sub> = 2.0 molal

TABLE VIII. QUALITATIVE ELECTROCHEMICAL TEST RESULTSa. Half Cell Potential of Anode1) Mg/BF<sub>3</sub>-Propylene carbonate ( $8.43 \times 10^{-4} \text{ ohm}^{-1}\text{cm}^{-1}$ )

Reference electrode: Ag-AgCl

OCV after abrasion of Mg: 1.74 - 1.82; 1.70

OCV after electrochemical reduction of Mg: 1.71; 1.90 - 1.92

	(approx. current density:	22 $\mu\text{a}/\text{cm}^2$	115 $\mu\text{a}/\text{cm}^2$	1.2 $\text{ma}/\text{cm}^2$
Discharge (	(voltage:	1.71	1.69	1.39

OCV after discharge:	1.71	1.71	1.71
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2) Mg/AlCl<sub>3</sub>-Propylene carbonate ( $6.16 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

Reference electrode: Ag-AgCl

OCV after abrasion of Mg: 1.70 - 1.78; 1.75 - 1.79

OCV after electrochemical reduction of Mg: 1.75 - 1.78; 1.75

	(approx. current density:	28 $\mu\text{a}/\text{cm}^2$	115 $\mu\text{a}/\text{cm}^2$	1.2 $\text{ma}/\text{cm}^2$	3.5 $\text{ma}/\text{cm}^2$
Discharge (	(voltage:	1.75-1.72	1.71-1.69	1.56	1.48

OCV after discharge:	1.73	1.71	1.72	1.75-1.71
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3) Mg/AlCl<sub>3</sub>-Benzonitrile

Reference electrode: Ag-AgCl

OCV after abrasion of Mg: 1.55

OCV after electrochemical reduction of Mg: 1.56 - 1.57

Note: Aqueous salt-acid immersion: 2.15 - stable 1.70 to 1.74 in 4 min.

4) Al/AlCl<sub>3</sub>-Benzonitrile

Reference electrode: Ag-AgCl

OCV after electrochemical reduction of Al: 0.84 dropped to 0.22

TABLE VIII. QUALITATIVE ELECTROCHEMICAL TEST RESULTS (Continued)a. Half Cell Potential of Anode (Continued)5) Mg/AlCl<sub>3</sub>-Dimethyl formamide ( $4.09 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

## a) Reference electrode: Cd

OCV after abrasion of Mg: 0.67 - 0.725

OCV after electrochemical reduction of Mg: 0.72 - 0.74

	(approx. current density: 28 $\mu\text{a}/\text{cm}^2$	100 $\mu\text{a}/\text{cm}^2$	1.85 $\text{ma}/\text{cm}^2$
Discharge (			
(voltage:	0.71	0.67-0.68	0.46-0.49

OCV after discharge: 0.725 0.72-0.74 0.74-0.80

Note: Near end of experiment - Cd gassing.

## b) Reference electrode: Ni

OCV after abrasion of Mg: 1.24 - 1.63

OCV after electrochemical reduction of Mg: 1.50 - 1.58

6) Mg/BF<sub>3</sub>-Dimethyl formamide ( $7 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

## Reference electrode: Cd

OCV after abrasion of Mg: 1.03 - 0.80

OCV after electrochemical reduction of Mg: 0.80

Note: Mg gassing.

7) Mg/LiCl-Dimethyl formamide ( $8.28 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

## a) Reference electrode: Cd

OCV after abrasion of Mg: 0.89 - 0.785; 0.85 - 0.765

OCV after electrochemical reduction of Mg: 2.04 - 1.98; 1.99 - 1.84

	(approx. current density: 60 $\mu\text{a}/\text{cm}^2$	240 $\mu\text{a}/\text{cm}^2$	1.3 $\text{ma}/\text{cm}^2$
Discharge (			
(voltage:	1.92-1.50	0.66-0.69	0.60-0.66

OCV after discharge: 1.80-1.82 1.06-1.60 0.85-1.00

Note: OCV after overnight standing = 0.485

TABLE VIII. QUALITATIVE ELECTROCHEMICAL TEST RESULTS (Continued)a. Half Cell Potential of Anode (Continued)7) Mg/LiCl-Dimethyl formamide ( $8.28 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

b) Reference electrode: Ni

OCV after abrasion of Mg: 1.18 - 1.55

OCV after electrochemical reduction of Mg: 2.81 - 2.85

Note 1: Mg gassing after overnight standing.

Note 2: Electrochemical reduction =  $2.7 \text{ ma/cm}^2$  for 20 min.8) Mg/MgCl<sub>2</sub>-Dimethyl formamide ( $4.87 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

Reference electrode: Cd

OCV after abrasion of Mg: 0.78 - 0.93

OCV after electrochemical reduction of Mg: 1.33 - 0.85

	(approx. current density:	63 $\mu\text{a/cm}^2$	305 $\mu\text{a/cm}^2$	1.1 $\text{ma/cm}^2$
Discharge (	(voltage:	0.80-0.875	0.1-0.85	0.5-0.785

OCV after discharge:	0.92-0.875	0.99-0.93	1.3-0.88
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9) Mg/BF<sub>3</sub>-Benzonitrile ( $1 \times 10^{-4} \text{ ohm}^{-1}\text{cm}^{-1}$ )

Reference electrode: Cd

OCV after abrasion of Mg: 0.88 - 1.045

OCV after electrochemical reduction of Mg: 1.11 - 1.15

	(approx. current density:	68 $\mu\text{a/cm}^2$	330 $\mu\text{a/cm}^2$
Discharge (	(voltage:	0.955-0.94	0.10-0.12

OCV after discharge:	1.19 -1.22	1.26-1.27
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Note: Black deposit on Mg electrodes.



TABLE VIII. QUALITATIVE ELECTROCHEMICAL TEST RESULTS (Continued)a. Half Cell Potential of Anode (Continued)10) Mg/KI-Dimethyl formamide ( $1.96 \times 10^{-2} \text{ ohm}^{-1}\text{cm}^{-1}$ )

Reference electrode: Cd

OCV after abrasion of Mg: 0.47 - 0.31

OCV after electrochemical reduction of Mg: 0.43 - 0.31

(approx. current density:  $70 \mu\text{a}/\text{cm}^2$ )  
 Discharge (  
 (voltage: 0.08-0.12

OCV after discharge: 0.52-0.40

11) Mg/ $\text{BCl}_3$ -Dimethyl formamide ( $6.13 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

a) Reference electrode: Cd

OCV after abrasion of Mg: 0.79 - 0.80

OCV after electrochemical reduction of Mg: 0.87

Note: Mg and Cd gassing. Cd darkened.

b) Reference electrode: Ni

OCV after abrasion of Mg: 1.70 - 1.69

OCV after electrochemical reduction of Mg: 1.71

(approx. current density:  $390 \mu\text{a}/\text{cm}^2$   $1.73 \text{ ma}/\text{cm}^2$ )  
 Discharge (  
 (voltage: 1.70 1.64

OCV after discharge: 1.71 1.71

Note: Mg gassing.

12) Mg/LiCl-Propylene carbonate ( $3.46 \times 10^{-4} \text{ ohm}^{-1}\text{cm}^{-1}$ )

Reference electrode: Ag-AgCl

OCV after abrasion of Mg: 1.39 - 1.50

OCV after electrochemical reduction of Mg: 2.20 - 1.39

(approx. current density:  $200 \mu\text{a}/\text{cm}^2$ )  
 Discharge (  
 (voltage: 0.70-1.21

OCV after discharge: 1.54-1.32

Note: Small granular black deposit on Mg electrodes.

TABLE VIII. QUALITATIVE ELECTROCHEMICAL TEST RESULTS (Continued)a. Half Cell Potential of Anode (Continued)13) Mg/LiCl-AlCl<sub>3</sub>-Propylene carbonate ( $6.75 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

Reference electrode: Ag-AgCl

OCV after abrasion of Mg: 1.76 - 1.77

OCV after electrochemical reduction of Mg: 1.75 - 1.74

	(approx. current density: 110 $\mu\text{a}/\text{cm}^2$	220 $\mu\text{a}/\text{cm}^2$	440 $\mu\text{a}/\text{cm}^2$
Discharge (			
(voltage:	1.66-1.67	1.54-1.56	1.11-1.41

OCV after discharge: 1.73 1.72-1.73 1.73

Note 1: No change in electrode appearance after overnight on open circuit.

Note 2: LiCl-AlCl<sub>3</sub>-PC: 22g. AlCl<sub>3</sub> in 200 cc. PC and saturated with LiCl.14) Mg/KI-Propylene carbonate ( $5.04 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

a) Reference electrode: Ag-AgCl

OCV after abrasion of Mg: 0.73 - 0.79

OCV after electrochemical reduction of Mg: 1.21 - 0.91

	(approx. current density: 115 $\mu\text{a}/\text{cm}^2$
Discharge (	
(voltage:	0.55-0.725

OCV after discharge: 0.86-0.84

Note: Mg anode gassing.

b) Reference electrode: Ag foil

OCV after abrasion of Mg: 0.775 - 0.715

OCV after electrochemical reduction of Mg: 1.28 - 0.65+

TABLE VIII. QUALITATIVE ELECTROCHEMICAL TEST RESULTS (Continued)a. Half Cell Potential of Anode (Continued)15) Mg/BCl<sub>3</sub>-Propylene carbonate ( $2.0 \times 10^{-3} \text{ ohm}^{-1}\text{cm}^{-1}$ )

Reference electrode: Ag-AgCl

OCV after abrasion of Mg: 2.09 - 2.08

OCV after electrochemical reduction of Mg: 2.07

	(approx. current density:	1.1 ma/cm <sup>2</sup>	2.8 ma/cm <sup>2</sup>
Discharge (	(voltage:	2.01	1.93-1.94

OCV after discharge: 2.05-2.04 2.04

Note: Mg gassing.

Note: with reference to the foregoing table:

- 1) OCV = open circuit voltage (reference vs. anode).
- 2) All voltages are in volts.
- 3) Area of Mg anode: approximately 3 cm x 0.3 cm.
- 4) Electrochemical reduction:  $100 \mu\text{a}/\text{cm}^2$  for 30 min.

Theoretical Potentials of the Reactions Study

<u>Reactions</u>	<u>Theoretical E° (volts)</u>
Mg + 2AgCl = MgCl <sub>2</sub> + 2Ag	1.93
Mg + CdCl <sub>2</sub> = MgCl <sub>2</sub> + Cd	1.29
Mg + NiCl <sub>2</sub> = MgCl <sub>2</sub> + Ni	1.65
Mg + CdF <sub>2</sub> = MgF <sub>2</sub> + Cd	1.98
Mg + 2AgI = MgI <sub>2</sub> + 2Ag	1.52
Mg + CdI <sub>2</sub> = MgI <sub>2</sub> + Cd	0.82
Al + 3AgCl = AlCl <sub>3</sub> + 3Ag	1.06

TABLE VIII. QUALITATIVE ELECTROCHEMICAL TEST RESULTS (Continued)b. Cell Potential

<u>Reaction</u>	<u>E<sup>0</sup></u>
$\text{Mg} + \text{CuF}_2 = \text{MgF}_2 + \text{Cu}$	2.92 Volts
$\text{Mg} + \text{NiF}_2 = \text{MgF}_2 + \text{Ni}$	2.21 Volts

1) Mg/KPF<sub>6</sub>-Propylene Carbonate (Sat.)/CuF<sub>2</sub>-Cu

Initial OCV: rises to 1.36 volts in 10 minutes

OCV after charge at 36  $\mu\text{a}/\text{cm}^2$  for 6 min.: 1.38 volts

OCV after discharge at 20  $\mu\text{a}/\text{cm}^2$  for 70 min.: 0.84 volts

OCV after charge at 40  $\mu\text{a}/\text{cm}^2$  for 20 min.: 1.34 rises to 1.44 volts in 6 hrs.

OCV after discharge at 30  $\mu\text{a}/\text{cm}^2$  for 20 min.: 1.30 rises to 1.43 volts in 87 hrs.

2) Mg/NaBF<sub>4</sub>-Propylene Carbonate (Sat.)/CuF<sub>2</sub>-Cu

Initial OCV: rises to 1.31 volts in 20 minutes

OCV after discharge at 90  $\mu\text{a}/\text{cm}^2$  for 30 min.: 1.31 volts

OCV after charge at 50  $\mu\text{a}/\text{cm}^2$  for 25 min.: 3.0 falls to 0.98 volts in 10 min.

OCV after charge at 1  $\text{ma}/\text{cm}^2$  for 50 min.: 2.56 falls to 2.07 volts in 10 min.

OCV after charge at 1  $\text{ma}/\text{cm}^2$  for 90 min.: 2.75 falls to 2.30 volts in 10 min.

OCV after charge at 1  $\text{ma}/\text{cm}^2$  for 80 min.: 2.8 falls to 2.31 volts in 10 min.

3) Mg/KPF<sub>6</sub>-Propylene Carbonate (Sat.)/NiF<sub>2</sub>-Ni

Initial OCV: rises to 1.24 volts in 30 minutes

OCV after discharge at 18  $\mu\text{a}/\text{cm}^2$  for 10 min.: 1.21 rises to 1.28 volts in 25 min.

OCV after charge at 2  $\text{ma}/\text{cm}^2$  for 10 min.: 2.98 falls to 2.7 volts in 4 min.

OCV after charge at 2  $\text{ma}/\text{cm}^2$  for 20 min.: 3.08 falls to 2.7 volts in 14 min.

OCV after charge at 2  $\text{ma}/\text{cm}^2$  for 30 min.: 3.10 falls to 2.7 volts in 22 min.

OCV after charge at 2  $\text{ma}/\text{cm}^2$  for 70 min.: 3.14 falls to 3.12 volts in 1 min.

TABLE VIII. QUALITATIVE ELECTROCHEMICAL TEST RESULTS (Continued)

b. Cell Potential (Continued)

4) Mg/NaBF<sub>4</sub>-Propylene Carbonate (Sat.)/NiF<sub>2</sub>-Ni

Initial OCV: rises to 1.24 volts in 20 minutes

OCV after discharge at 40  $\mu$ a/cm<sup>2</sup> for 10 min.: 1.31 falls to 1.27 volts in 10 min.

OCV after charge at 1 ma/cm<sup>2</sup> for 10 min.: 2.15 falls to 1.44 volts in 10 min.

OCV after charge at 0.3 ma/cm<sup>2</sup> for 60 min.: 2.32 falls to 1.71 volts in 10 min.

OCV after charge at 0.2 ma/cm<sup>2</sup> for 160 min.: 2.9 falls to 1.85 volts in 10 min.

OCV after charge at 0.25 ma/cm<sup>2</sup> for 110 min.: 2.03 volts

OCV after discharge at 25  $\mu$ a/cm<sup>2</sup> for 20 min.: 1.37 rises to 1.44 volts in 3 min.

5) Mg/KPF<sub>6</sub>-Dimethyl Formamide (0.79 molal)/NiF<sub>2</sub>-Ni

Initial OCV: 1.20 volts

OCV after discharge at 30  $\mu$ a/cm<sup>2</sup> for 10 min.: 1.06 rises to 1.16 volts in 30 min.

OCV after charge at 2 ma/cm<sup>2</sup> for 10 min.: 3.0 falls to 1.48 volts in 2 min.

OCV after charge at 1 ma/cm<sup>2</sup> for 40 min.: 3.1 falls to 1.68 volts in 2 min.

OCV after charge at 1 ma/cm<sup>2</sup> for 300 min.: 3.26 falls to 2.47 volts in 2 min.

6) Mg/NaBF<sub>4</sub>-DMF (0.68 molal)/NiF<sub>2</sub>-Ni

Initial OCV: 1.22 volts

OCV after discharge at 30  $\mu$ a/cm<sup>2</sup> for 10 min.: 1.23 falls to 1.13 volts in 10 min.

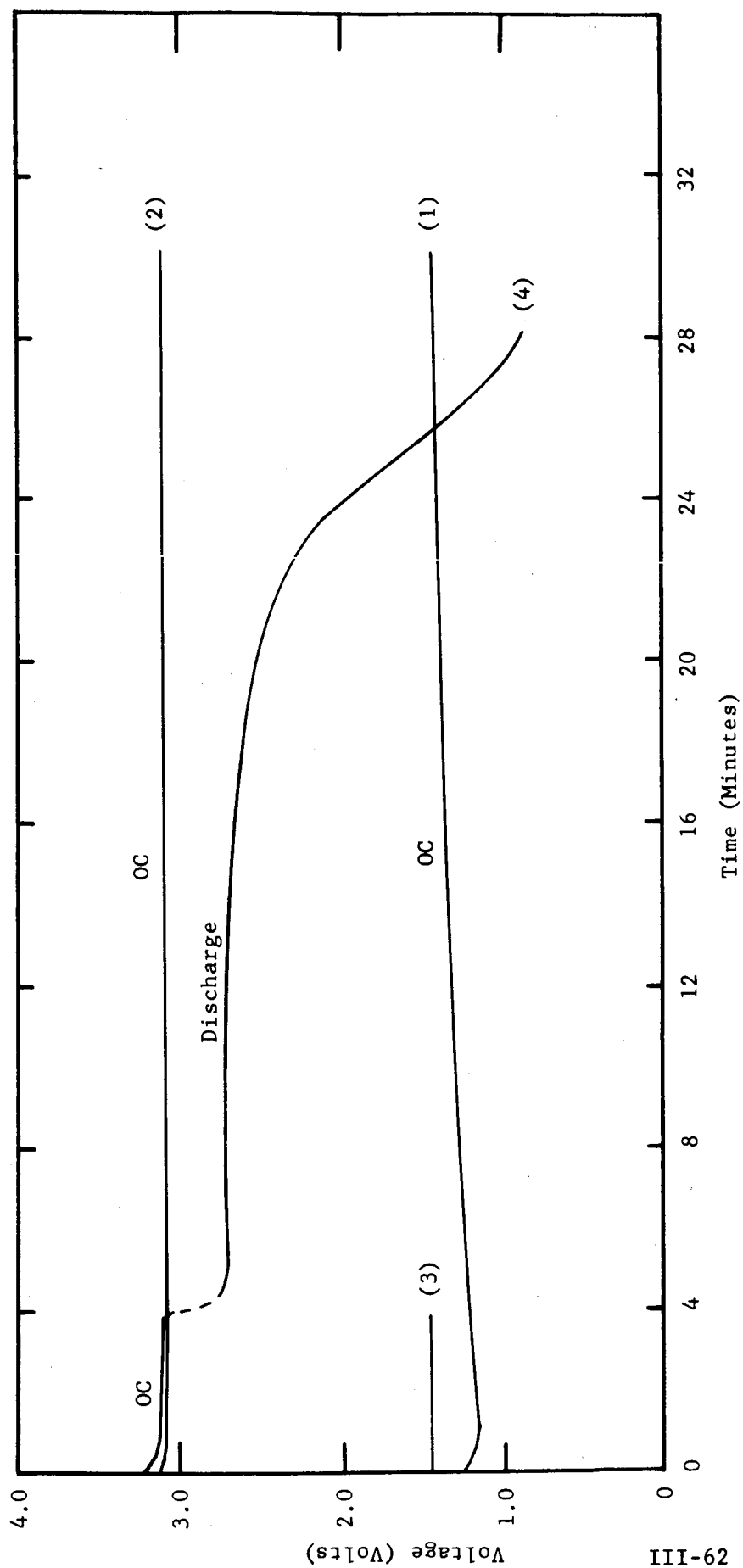
OCV after charge at 1 ma/cm<sup>2</sup> for 10 min.: 2.18 falls to 1.89 volts in 1 min.

OCV after charge at 1 ma/cm<sup>2</sup> for 70 min.: 2.22 falls to 1.80 volts in 3 min.

OCV after charge at 0.3 ma/cm<sup>2</sup> for 17 hr.: 2.42 falls to 1.91 volts in 3 min.

OCV after discharge at 10  $\mu$ a/cm<sup>2</sup> for 5 min.: 1.06 rises to 1.52 volts in 160 min.

Note: All current densities are approximate average values and are based on Mg anode area.



- (1)  $\text{NiF}_2$ -Ni cathode vs. Mg anode on O.C. before charge.
- (2) Cathode vs. anode on O.C. after  $\ll 1 \text{ ma/cm}^2$  charge for 15 hours.
- (3) Cathode after charge vs. fresh Mg strip.
- (4) After charged at  $11 \text{ ma/cm}^2$  for 30 minutes, cathode vs. anode on discharge ( $140\text{--}100 \mu\text{a/cm}^2$ ).

FIGURE 10. CELL POTENTIAL OF  $\text{Mg/KPF}_6$ -  
PROPYLENE CARBONATE (0.96 molal)/ $\text{NiF}_2$ - $\text{Ni}$

TABLE IX. STABILITY OF ELECTRODE MATERIALS IN  
SOLVENTS AND ELECTROLYTES AT ROOM TEMPERATURE

	<u>Page No.</u>
a. In Dimethylformamide . . . . .	III-64
b. In $\text{AlCl}_3$ -Dimethylformamide . . . . .	III-65
c. In $\text{BF}_3$ -Dimethylformamide . . . . .	III-65
d. In Propylene Carbonate . . . . .	III-66
e. In $\text{BF}_3$ -Propylene Carbonate . . . . .	III-67
f. In Benzonitrile . . . . .	III-68
g. In $\text{AlCl}_3$ -Benzonitrile . . . . .	III-69
h. In $\text{BF}_3$ -Benzonitrile . . . . .	III-69

TABLE IX. STABILITY OF ELECTRODE MATERIALS IN  
SOLVENTS AND ELECTROLYTES AT ROOM TEMPERATURE

a. In Dimethylformamide		Gm. Sample Before Exposure	After Exposure	Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Length of Test (Hrs.)	Visual Observation
1.	Blank	---	---	1.78 x 10 <sup>-6</sup> 1.87 x 10 <sup>-6</sup>	0 72	--- ---
	Al	0.0102	0.0102	2.03 x 10 <sup>-6</sup>	72	No apparent reaction
	Co	0.1552	0.1552	1.97 x 10 <sup>-6</sup>	72	No apparent reaction
	Cu	0.6405	0.6407	3.79 x 10 <sup>-6</sup>	72	No apparent reaction
	Mg	0.0421	0.0416	3.35 x 10 <sup>-6</sup>	72	No apparent reaction
	Ni	0.6890	0.6895	1.94 x 10 <sup>-6</sup>	72	No apparent reaction
2.	Blank	---	---	1.55 x 10 <sup>-6</sup> 2.03 x 10 <sup>-6</sup>	0 23	--- ---
	CoF <sub>3</sub>	---	---	2.45 x 10 <sup>-4</sup>	23	Light brown
	CuF <sub>2</sub>	---	---	6.27 x 10 <sup>-5</sup>	23	No apparent reaction
	NiCl <sub>2</sub> · 6H <sub>2</sub> O (dried)	---	---	2.31 x 10 <sup>-3</sup>	23	Blue green
	NiF <sub>2</sub>	---	---	8.80 x 10 <sup>-5</sup>	23	No apparent reaction



TABLE IX. STABILITY OF ELECTRODE MATERIALS IN  
SOLVENTS AND ELECTROLYTES AT ROOM TEMPERATURE (Continued)

b. In $\text{AlCl}_3$ -Dimethylformamide					
	Gm. Sample		Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Length of Test (Hrs.)	Visual Observation
	Before Exposure	After Exposure			
Blank	---	---	$1.32 \times 10^{-2}$ $4.03 \times 10^{-3}$	0 30	--- ---
$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (dried)	---	---	$2.90 \times 10^{-3}$	30	Blue -- Dark blue
c. In $\text{BF}_3$ -Dimethylformamide					
	Gm. Sample		Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Length of Test (Hrs.)	Visual Observation
	Before Exposure	After Exposure			
Blank	---	---	$8.90 \times 10^{-3}$ $1.34 \times 10^{-2}$	0 146	--- ---
$\text{CoF}_3$	---	---	$1.62 \times 10^{-2}$	146	Light brown -- Purple
$\text{CuF}_2$	---	---	$1.45 \times 10^{-2}$	146	Blue -- Blue green
$\text{NiF}_2$	---	---	$1.30 \times 10^{-2}$	146	Light green

TABLE IX. STABILITY OF ELECTRODE MATERIALS IN  
SOLVENTS AND ELECTROLYTES AT ROOM TEMPERATURE (Continued)

d. In Propylene Carbonate						
	Gm. Sample		Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Length of Test (Hrs.)		Visual Observation
	Before Exposure	After Exposure				
1. Blank	---	---	1.15 x 10 <sup>-5</sup> 1.16 x 10 <sup>-5</sup>	0 72	---	---
CuF <sub>2</sub>	---	---	5.20 x 10 <sup>-5</sup>	72	No apparent reaction	No apparent reaction
Mg	0.1488	0.1494	1.12 x 10 <sup>-5</sup>	72	No apparent reaction	No apparent reaction
2. Blank	---	---	1.15 x 10 <sup>-5</sup> 1.20 x 10 <sup>-5</sup>	0 94	---	---
CoF <sub>3</sub>	---	---	1.03 x 10 <sup>-4</sup>	94	Brown	Brown
NiCl <sub>2</sub> · 6H <sub>2</sub> O(dried)	---	---	1.53 x 10 <sup>-5</sup>	94	Green	Green
NiF <sub>2</sub>	---	---	7.25 x 10 <sup>-4</sup>	94	No apparent reaction	No apparent reaction

TABLE IX. STABILITY OF ELECTRODE MATERIALS IN  
SOLVENTS AND ELECTROLYTES AT ROOM TEMPERATURE (Continued)

e. In <u>BF<sub>3</sub>-Propylene Carbonate</u>		<u>Specific Conductance</u> (ohm <sup>-1</sup> cm <sup>-1</sup> )	<u>Length of</u> <u>Test (Hrs.)</u>	<u>Visual Observation</u>
<u>Gm. Sample</u>				
<u>Before</u> <u>Exposure</u>	<u>After</u> <u>Exposure</u>			
1. Blank	---	8.66 x 10 <sup>-4</sup>	0	---
		8.94 x 10 <sup>-4</sup>	72	---
CuF <sub>2</sub>	---	1.88 x 10 <sup>-3</sup>	72	No apparent reaction
Mg	0.1201	1.58 x 10 <sup>-3</sup>	72	Apparently soluble
2. Blank	---	8.42 x 10 <sup>-4</sup>	0	---
		1.29 x 10 <sup>-3</sup>	94	---
CoF <sub>3</sub>	---	1.37 x 10 <sup>-3</sup>	94	No apparent reaction
NiF <sub>2</sub>	---	1.72 x 10 <sup>-3</sup>	94	No apparent reaction

TABLE IX. STABILITY OF ELECTRODE MATERIALS IN  
SOLVENTS AND ELECTROLYTES AT ROOM TEMPERATURE (Continued)

f. In Benzonitrile

	Gm. Sample		Specific Conductance (ohm <sup>-1</sup> cm <sup>-1</sup> )	Length of Test (Hrs.)	Visual Observation
	Before Exposure	After Exposure			
1. Blank	---	---	1.92 x 10 <sup>-7</sup> 3.07 x 10 <sup>-7</sup>	0 144	--- ---
Al	0.1207	0.1206	2.50 x 10 <sup>-7</sup>	144	No apparent reaction
Co	0.1555	0.1555	3.09 x 10 <sup>-7</sup>	144	No apparent reaction
Cu	0.6703	0.6703	2.97 x 10 <sup>-7</sup>	144	No apparent reaction
Mg	0.0851	0.0850	2.90 x 10 <sup>-7</sup>	144	No apparent reaction
Ni	0.7077	0.7077	3.57 x 10 <sup>-7</sup>	144	No apparent reaction
2. *Blank	---	---	2.08 x 10 <sup>-7</sup> 2.26 x 10 <sup>-7</sup>	0 72	--- ---
*Li	---	---	6.59 x 10 <sup>-5</sup>	72	Brown viscous liquid
3. **Blank	---	---	1.59 x 10 <sup>-7</sup> 2.08 x 10 <sup>-7</sup>	0 114	--- ---
**CoF <sub>3</sub>	---	---	3.14 x 10 <sup>-5</sup>	114	Brown
**CuF <sub>2</sub>	---	---	6.42 x 10 <sup>-5</sup>	114	Brown
**NiCl <sub>2</sub> 6H <sub>2</sub> O(dried)	---	---	1.32 x 10 <sup>-6</sup>	114	No apparent reaction
**NiF <sub>2</sub>	---	---	9.11 x 10 <sup>-6</sup>	114	Gold color liquid

\* Dry argon atmosphere.

\*\* Dry atmosphere.

TABLE IX. STABILITY OF ELECTRODE MATERIALS IN  
SOLVENTS AND ELECTROLYTES AT ROOM TEMPERATURE (Continued)

g. In $\text{AlCl}_3$ -Benzonitrile					
	Gm. Sample		Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Length of Test (Hrs.)	Visual Observation
	Before Exposure	After Exposure			
Blank	---	---	$4.38 \times 10^{-3}$ $4.00 \times 10^{-3}$	0 72	--- ---
$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}(\text{dried})$	---	---	$4.15 \times 10^{-3}$	72	Brown
h. In $\text{BF}_3$ -Benzonitrile					
	Gm. Sample		Specific Conductance ( $\text{ohm}^{-1} \text{ cm}^{-1}$ )	Length of Test (Hrs.)	Visual Observation
	Before Exposure	After Exposure			
Blank	---	---	$9.14 \times 10^{-5}$ $1.01 \times 10^{-4}$	0 147	Brown Brown
$\text{CoF}_3$	---	---	$3.48 \times 10^{-4}$	147	Dark brown
$\text{CuF}_2$	---	---	$7.12 \times 10^{-4}$	147	Dark olive color
$\text{NiF}_2$	---	---	$2.72 \times 10^{-4}$	147	Brown purple

TABLE X. STABILITY OF SEPARATOR MATERIALS  
IN SOLVENTS AT ROOM TEMPERATURE

Separator Material	Grade No.	Visible Change After 30 Days	Initial Conductance of Solvent (ohm <sup>-1</sup> cm <sup>-1</sup> )	Conductance of Solvent After 30 Days (ohm <sup>-1</sup> cm <sup>-1</sup> )	Viscosity of Solvent After 30 Days (Centipoises)
a. In Dimethylformamide					
Blank	---	---	8.25 x 10 <sup>-7</sup>	9.42 x 10 <sup>-7</sup>	0.91
Cotton	R2205	Stickiness; Swelling	8.49 x 10 <sup>-7</sup>	2.69 x 10 <sup>-6</sup>	0.89
Viscose	M1216	Stickiness	8.25 x 10 <sup>-7</sup>	---	0.98
Polypropylene	EM476	None	8.39 x 10 <sup>-7</sup>	3.00 x 10 <sup>-6</sup>	0.97
Polypropylene	SM91	None	8.40 x 10 <sup>-7</sup>	2.54 x 10 <sup>-6</sup>	0.93
Dacron	M1481	None	8.25 x 10 <sup>-7</sup>	---	0.91
Dacron	M1432	None	8.25 x 10 <sup>-7</sup>	2.18 x 10 <sup>-6</sup>	0.89
Dacron	EM373	Stickiness	8.25 x 10 <sup>-7</sup>	3.00 x 10 <sup>-6</sup>	0.91
b. In Propylene Carbonate					
Blank	---	---	1.25 x 10 <sup>-5</sup>	1.34 x 10 <sup>-5</sup>	2.39
Cotton	R2205	Some Swelling	1.24 x 10 <sup>-5</sup>	1.35 x 10 <sup>-5</sup>	2.39
Polypropylene	EM476	None	1.25 x 10 <sup>-5</sup>	1.46 x 10 <sup>-5</sup>	2.45
Polypropylene	SM91	None	1.25 x 10 <sup>-5</sup>	1.41 x 10 <sup>-5</sup>	2.46

TABLE X. STABILITY OF SEPARATOR MATERIALS  
IN SOLVENTS AT ROOM TEMPERATURE (Continued)

<u>Separator Material</u>	<u>Grade No.</u>	<u>Visible Change After 30 Days</u>	<u>Initial Conductance of Solvent (ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Conductance of Solvent After 30 Days (ohm<sup>-1</sup> cm<sup>-1</sup>)</u>	<u>Viscosity of Solvent After 30 Days (Centipoises)</u>
b. <u>In Propylene Carbonate</u> (Continued)					
Dacron	M1481	None	1.25 x 10 <sup>-5</sup>	1.36 x 10 <sup>-5</sup>	2.44
Dacron	M1432	None	1.25 x 10 <sup>-5</sup>	1.35 x 10 <sup>-5</sup>	2.39
Dacron	EM372	Stickiness	1.25 x 10 <sup>-5</sup>	1.42 x 10 <sup>-5</sup>	2.51

#### IV. APPENDIXES



## APPENDIX I.

### ENERGY DENSITY CURVES

A simplified family of curves has been found for the interrelationship between cell voltage, equivalent weight of reacting materials, and energy density (Figure 1, page III-5). The curves are based on the thermodynamic equation,

$$(1) \Delta F_r = -nFE_c$$

where  $\Delta F_r$  = free energy of reaction expressed as watt-hours per pound

$n$  = the number of g. equivalents per pound

$F$  = Faraday constant expressed in ampere hours per g. equivalent

$E_c$  = cell voltage

To obtain a more useful form of this equation, we have related  $n$  to the equivalent weight in grams ( $W_{eq}$ ),

$$n = \frac{\text{g. equivalents}}{\text{pound}} = \frac{453.6 \text{ g/lb.}}{W_{eq} \text{ g/g-equiv.}}$$

and  $F$  is expressed as  $26.8 \frac{\text{Ampere Hours}}{\text{g. equivalent}}$

Equation (1) can then be rewritten:

$$(2) \Delta F_r \frac{(\text{watt-hours})}{(\text{pound})} = \frac{-453.6 \text{ g/lb.}}{W_{eq}} \times 26.8 \frac{\text{AH}}{\text{g. equiv.}} \times E_c (\text{volts})$$

If the constants are multiplied we obtain:

$$-C = -453.6 \text{ g/lb.} \times 26.8 \frac{\text{AH}}{\text{g. equiv.}} = -12,156.5 \frac{\text{g-AH}}{\text{lb.-g. equiv.}}$$

Equation (2) then becomes:

$$(3) \Delta F_r \frac{(\text{watt-hours})}{(\text{pound})} = \frac{C}{W_{eq} \frac{(\text{grams})}{\text{g/eq.}}} \times E_c (\text{volts})$$

Finally, rearrangement of (3) gives the equation:

$$(4) E_c (\text{volts}) = \frac{(\Delta F_r)}{(C)} W_{eq} (\text{grams})/\text{g. equiv.}$$

This equation is equivalent to  $y = mx$  (where  $y = E_c$ ,  $x = W_{eq}$ ,  $m = \frac{(\Delta F_r)}{(C)}$ ), which is the equation of a straight line passing through the origin. The slope,  $m$ ,

of the line is  $\frac{\Delta F_r}{C}$ . Since C is a constant, it is obvious that the free energy of reaction (watt-hours per pound) must determine the slope of the line. Thus, all points on the same line will have the same energy density. Any point (x, y) is specific for a given electrode couple, and the point also establishes the energy density of the couple. All couples on or above the 500 watt-hour per pound line possess sufficient energy density to be considered in our screening program.

A three digit code number was used to identify the anode-cathode couples shown as circles on the figure, but no explanation of the meaning of code numbers was given. Therefore, we describe these code numbers below.

The first number designates the anode material (e.g., Mg = 1). The second number - i.e., the middle number - designates the metallic part of cathode material (e.g., Co = 4). The third number combined with the second number reveals the identity of the cathode material and also the relative theoretical energy density of the anode-cathode couple (e.g., fluoride = 3 to 5; 3 for the highest energy density fluoride couple, 4 for the next highest energy density fluoride couple, etc.). Thus, magnesium-cobalt fluoride systems are designated by numbers 143, 144 and 145, and the energy densities of these systems are in the following order: 143 > 144 > 145.

<u>Code No.</u>	<u>Watt-hours/lb.</u>	<u>Reaction</u>
143	550	$3\text{Mg} + 2\text{CoF}_3 \rightarrow 3\text{MgF}_2 + 2\text{Co}$
144	454	$\text{Mg} + \text{CoF}_2 \rightarrow \text{MgF}_2 + \text{Co}$
145	427	$\text{Mg} + 2\text{CoF}_3 \rightarrow \text{MgF}_2 + 2\text{CoF}_2$

The applicable chemical equations, energy densities, and the corresponding code numbers are presented in Table I.

APPENDIX II.

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***Session on Primary Batteries***

**INVESTIGATIONS OF HIGH ENERGY DENSITY  
ELECTROCHEMICAL SYSTEMS**

**Part 1 — Electrolytes**

**W. E. Elliott, S. L. Hsu, W. I. Towle  
Globe-Union Inc.**

***Introduction:***

The objective of this research program is to develop a primary battery with an energy density of 200 watt-hours per pound of total battery weight. The work is conducted under the sponsorship of the National Aeronautics and Space Administration, Lewis Research Center, on contract NAS3-2790.

Commercial primary battery electrode couples do not possess sufficiently high theoretical free energy densities to

meet the objectives of such a program. The inability to realize 100% of the energy from the active material and the presence of such "dead weight" items as separators, case, electrolyte, etc. usually result in an energy yield per pound of 40% or less of the theoretical free energy density of the electrode couple. Thus a minimum value of 500 watt-hours per pound based on active material alone will be required for a real system to yield 200 watt-hours per pound. This requirement immediately

limits the suitable electrode materials to those of low equivalent weight and high energy. If the anodes are to be metals, they must be selected from the upper left-hand corner of the periodic table, groups IA and IIA. Cathode depolarizers should also have low equivalent weight and in general will consist of two or more elements selected from the right-hand side of the periodic table. Some examples of couples which meet the theoretical energy requirements are shown in Table 1.

TABLE I  
HIGH ENERGY DENSITY ELECTRODE COUPLES  
AT 25°C.

REACTION <sup>1</sup>					E°	WH/LB
2 Li +	CuF <sub>2</sub> =	2 LiF +	Cu		3.55	749
2 Li +	NiF <sub>2</sub> =	2 LiF +	Ni		2.83	620
2 Li +	NiCl <sub>2</sub> =	2 LiCl +	Ni		2.57	437
3 Li +	CoF <sub>3</sub> =	3 LiF +	Co		3.64	965
Li +	CoF <sub>3</sub> =	LiF +	CoF <sub>2</sub>		5.14	510
2 Li +	CoF <sub>2</sub> =	2 LiF +	Co		2.88	633
2 Li +	CuO =	Li <sub>2</sub> O +	Cu		2.25	587
2 Li +	NiO =	Li <sub>2</sub> O +	Ni		1.79	492
Mg +	CuF <sub>2</sub> =	MgF <sub>2</sub> +	Cu		2.92	566
Mg +	NiF <sub>2</sub> =	MgF <sub>2</sub> +	Ni		2.21	445
3 Mg +	2CoF <sub>3</sub> =	3 MgF <sub>2</sub> +	2Co		2.89	691
Mg +	CuO =	MgO +	Cu		2.30	538
Mg +	NiO =	MgO +	Ni		1.83	451
Mg +	AgO =	MgO +	Ag		2.98	491
Ca +	CuF <sub>2</sub> =	CaF <sub>2</sub> +	Cu		3.51	604
Ca +	NiF <sub>2</sub> =	CaF <sub>2</sub> +	Ni		2.82	501
Ca +	CuO =	CaO +	Cu		2.47	503

The use of highly active metal anodes precludes the use of aqueous electrolytes because reaction with water would result in wasteful corrosion of the metal, hydrogen gas evolution, and reduced electrode potentials. Thus, whatever solvents are used, they should preferably be aprotic in nature.

Referring again to the figure of 500 watt-hours per pound, the use of this value implies also that the polarization and IR loss in the new systems will be roughly comparable with those in existing systems. Figure 1 illustrates the importance of electrolyte conductivity on IR loss. Since the electrolyte not only thus determines IR loss but may influence concentration and activation polarization as well, its importance as a key factor in overall battery performance is readily recognized. Because the nature of concentrated nonaqueous electrolytes is not sufficiently well understood,<sup>2,3</sup> our investigation up to this time has been primarily directed toward a clarification of the basic factors influencing their behavior, and has been based to some extent on data obtained by others<sup>4</sup>. The ultimate goal of this part of the program is to achieve a substantially improved nonaqueous electrolyte with electrical conductivity hopefully approaching that of aqueous systems.

#### Aqueous Electrolytes:

As a reference point, many aqueous solutions possess properties which are required for a good battery electrolyte. In considering the properties which make water a particularly good solvent for such service, we note the following:<sup>2,3</sup>

It forms hydrogen bonds, which contribute to a high di-

electric constant, thus facilitating the dissolution and separation of positive and negative ions. It forms coordination complexes with positive ions, thus providing them with a sheath which may further promote dissolution and separation of ions by reduction in charge concentration. It has a low viscosity, allowing high ion mobility. It has a low molar volume which permits more molecules of high dielectric constant in the space between ions and thus helps minimize ionic in-

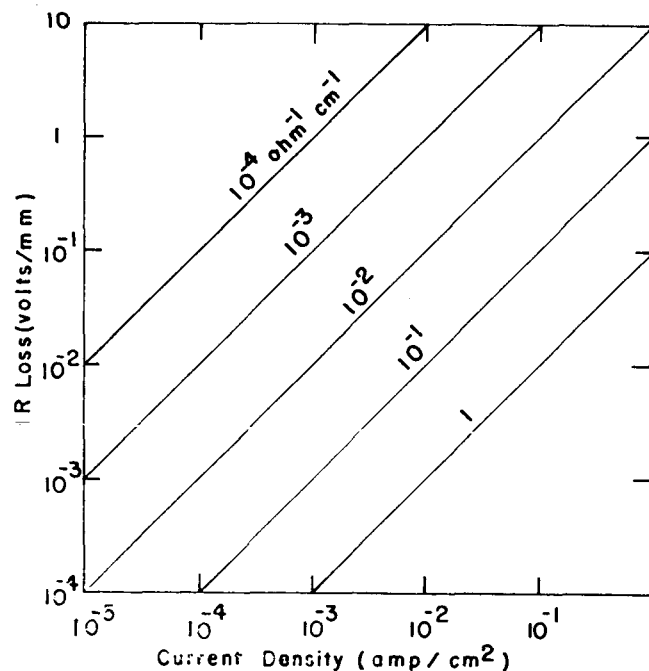


Figure 1. The effect of the specific conductance on IR losses at an electrode spacing of 1 mm.

teractions which reduce the conductance and mobility of the ions. Water also possesses the property of allowing exceptionally high hydrogen and hydroxyl ion mobility due to quantum mechanical effects. It has a wide liquidus range, particularly with dissolved ionic substances. The effect of this combination of properties is that high solubility and a high degree of dissociation are commonly observed with ionic materials in aqueous solution.

For reference it is now of interest to observe the characteristics of aqueous electrolytes, as summarized in Table II. Important points to note are:

- 1) The limiting equivalent conductance values,  $\Lambda^*$ , all fall within a half an order of magnitude, even when the hydrogen ion or hydroxyl ion is present.
- 2) The range of equivalent conductance,  $\Lambda$ , at maximum conductance is even narrower than that of the limiting equivalent conductance except for the weak electrolytes.
- 3) The concentration of each solute at maximum conductance is quite high, 3-6 molal, yet the apparent degree of dissociation,  $\Lambda/\Lambda^*$ , generally exceeds 25%.
- 4) The maximum specific conductance,  $L$ , of most strong electrolytes in water falls into the range of  $10^{-1}$  to  $1 \text{ ohm}^{-1} \text{ cm}^{-1}$ .

A feature of the aqueous solutions is the decrease in the apparent degree of dissociation with increasing concentration. Ion pair formation, multiple ion formation, the relaxation ef-

TABLE II  
CONDUCTANCE IN AQUEOUS SOLUTIONS AT 18°C.

SOLUTE	$\Lambda^\circ$ <sup>2</sup>	$L_{\max}$ <sup>5</sup>	$C_{L\max}$ <sup>5</sup>	$\Lambda_{\max}$ <sup>5</sup>	$\Lambda_{\max} / \Lambda^\circ$
H <sub>2</sub> SO <sub>4</sub>	383.4	0.74	4.4	98.9	0.26
HCl-15°C	362	0.76	6.9	126.2	0.35
NaOH	213.8	0.35	4.4	80.0	0.37
KOH-15°C	220	0.54	6.0	96.8	0.44
KCl	129.9	0.28	3.6	87.5	0.67
LiCl	98.8	0.17	5.9	31.9	0.32
KI	130.4	0.43	7.4	78.2	0.60
NH <sub>4</sub> Cl	129.9	0.40	6.2	80.5	0.62
NaAc	77.8	0.065	3.0	24.2	0.27
HAc	350	0.0016	4.2	0.47	0.0013
NH <sub>3</sub> -15°C	220	0.0011	2.4	0.475	0.0022

fect and the electrophoretic effect all restrict the specific conductance by interfering<sup>2,3</sup> with the freedom and mobility of the ions. An indication of the effect of these interactions is shown by the increased viscosity which can be attributed to the larger aggregates of solutes or solvated groups as the concentrations are increased. The net effect of the sum of these interactions is demonstrated by Figure 2, showing con-

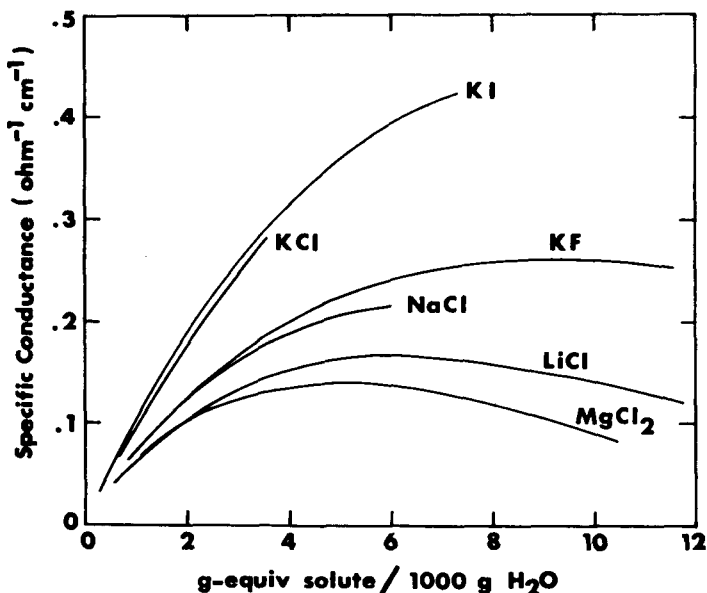


Figure 2. The effect of solubility and ion size on the conductance of aqueous solutions at 18°C.

centration vs. specific conductance for aqueous solutions. It is noteworthy that many of the curves pass through a maximum, particularly where solubility is high. Thus it appears that water might perform even better as a solvent if it were not subject to these interactions.

#### Nonaqueous Electrolytes

The limiting equivalent conductances of some nonaqueous electrolytes are compared to those of aqueous electrolytes in Table III. Certain aspects of the data are noteworthy.

1) The solvent appears to control the  $\Lambda^\circ$  value regardless of the solute used.

- 2) The dielectric constant does not of itself dominate the  $\Lambda^\circ$  values.
- 3) The viscosity of the solvents is very important. This can be observed by comparison of the  $\Lambda^\circ$  values of the low viscosity liquids with those of high viscosity. If Walden's rule ( $\Lambda^\circ \eta = \text{constant}$ ) is applied to the average values of  $\Lambda^\circ$  for all the solutes in a given solvent, a remarkably high degree of uniformity is observed.

TABLE III  
THE COMPARISON OF LIMITING EQUIVALENT CONDUCTANCES OF AQUEOUS AND NONAQUEOUS ELECTROLYTES AT 25°C.

	H <sub>2</sub> O <sup>2</sup>	CH <sub>3</sub> OH <sup>2</sup>	HCN <sup>2</sup> (18°C)	DMF <sup>2</sup>	FA <sup>2</sup>	NMF <sup>2</sup>
	$\epsilon=78.3$ $\eta=0.89$	$\epsilon=32.6$ $\eta=0.54$	$\epsilon=118.2$ $\eta=0.206$	$\epsilon=36.7$ $\eta=0.796$	$\epsilon=109.5$ $\eta=3.3$	$\epsilon=182.4$ $\eta=1.65$
LiCl	114.95	92.2	345.4			
NaCl	126.45	97.6				
KCl	149.85	104.8	363.4		29.85	41.9
KBr	151.7	108.95	363.2	84.1		43.7
KI	150.3	115.15	363.9	82.6	29.3	45.0
RbCl	154.15	108.6 *	363.2			
CsCl	153.55	113.6 *	368.2			
KCNS		114.5 *	358.0	90.3		
KNO <sub>3</sub>	144.96	114.5 *	353.9	88.1		
KClO <sub>4</sub>	140.8		353.3	82.8		
NaI	126.9	107.97		82.0	26.74	44.4
$\bar{\Lambda}_o$	141.4	107.8	359.2	85.0	28.6	43.8
$\bar{\Lambda}_o \eta$	125.8	58.2	75.4	67.6	94.5	72.2

#### Experimental

It is apparent from the above analysis that battery electrolytes will have to be at a relatively high concentration of solute. Only a limited amount of work has been done in this area in nonaqueous solvents. Therefore, the principal need for experimental data is to furnish information on principles governing conductance at higher concentration. To provide this, a broad examination of many solute-solvent systems is needed. In order to move rapidly, we chose to examine the conductivities of many mixtures of solutes and solvents in a one-molal ratio. That is, sufficient solute was used to produce a one-molal solution at complete solubility. We then simply noted whether the solute did or did not dissolve at this concentration. In most cases complete solution did not occur; thus the values obtained represent saturated solutions. Where complete solution occurred, the values are so indicated in the accompanying data.

Table IV illustrates the type of data obtained. The following points are noteworthy:

- 1) Contrary to the behavior in water, each of the three aprotic solvents tested yielded a range in conductivity covering several orders of magnitude. This is perhaps due to wide variations in solubility although quantitative data have not yet been obtained.
- 2) The solvent is a dominating factor in determining the overall level of conductivity.
- 3) The quarternary ammonium salt, tetrabutylammonium iodide, gives higher conductivities in the organic solvents than in water probably because of higher solubility, attributable perhaps to the solubilizing influence of the butyl groups, in the aprotic solvents.

4) Strikingly, the salts arrange themselves with rather few exceptions in the same order of conductivity in the three aprotic solvents.

5) It is also apparent that compounds with the largest ions lie at the top of the table while those containing at least one small ion are at the bottom of the table.

The influence of the solutes is demonstrated more graphically in Figure 3, showing solubility data in dimethylformamide.

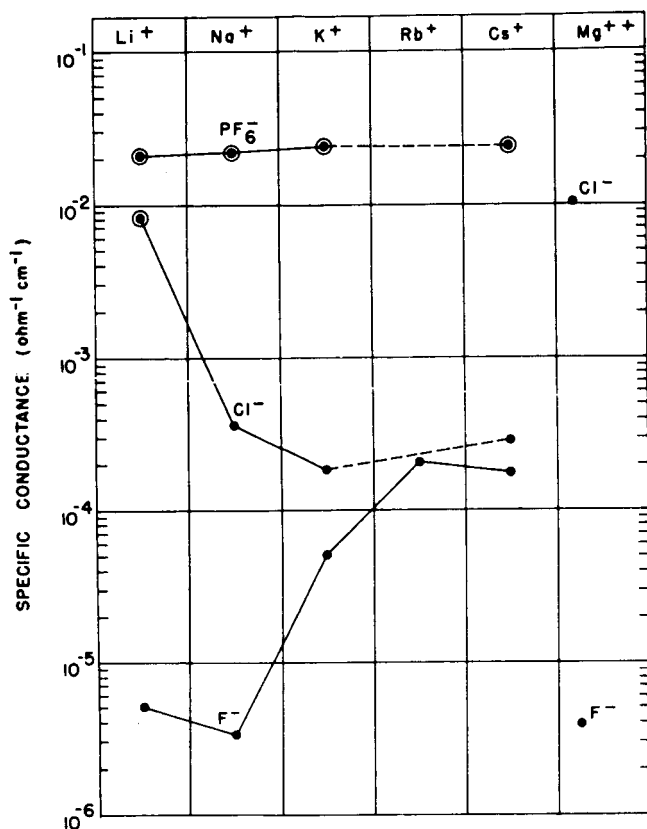


Figure 3. Effect of solutes on the specific conductance in N,N'-dimethyl formamide at 25°C.

Note: Encircled values are 1 molal solutions; all others became saturated solutions at less than a 1 molal concentration.

It appears that the anion size has a direct influence on solubility and conductivity.

Cation influence is apparently more complicated, possibly because of varying degrees of solvation. More quantitative data are needed for further clarification of the picture.

### Summary

The theoretical free energy density for suitable electrode couples should be at least 500 watt-hours per pound, even assuming that polarization and IR losses are as good as for the best existing systems. Since the highly active electrode materials thus required would decompose water, aprotic electrolytes are needed. Aqueous battery electrolytes possess conductances of approximately  $5 \times 10^{-1}$  to  $1 \text{ ohm}^{-1} \text{ cm}^{-1}$ , while

TABLE IV  
SPECIFIC CONDUCTANCE OF  
NONAQUEOUS ELECTROLYTES AT 25°C.

	WATER $\epsilon=78$ $\eta=0.89$	DMF $\epsilon=37$ $\eta=0.80$	PC $\epsilon=64$ $\eta=2.53$	BN $\epsilon=25$ $\eta=1.05$
KPF <sub>6</sub>	$4.4 \times 10^{-2}$	$2.5 \times 10^{-2}$	$7.8 \times 10^{-3}$	$3.6 \times 10^{-4}$
KI	$1.1 \times 10^{-1}$	$2.2 \times 10^{-2}$	$5.2 \times 10^{-3}$	$1.3 \times 10^{-4}$
TBAI	$2.2 \times 10^{-3}$	$1.1 \times 10^{-2}$	$4.5 \times 10^{-3}$	—
NaBF <sub>4</sub>	$6.5 \times 10^{-2}$	$1.0 \times 10^{-2}$	$1.1 \times 10^{-3}$	$3.8 \times 10^{-5}$
LiCl	$7.1 \times 10^{-2}$	$8.3 \times 10^{-3}$	$3.4 \times 10^{-4}$	$1.4 \times 10^{-6}$
BeCl <sub>2</sub>	HYDROLYZES	$2.1 \times 10^{-3}$	$5.0 \times 10^{-4}$	$5.7 \times 10^{-5}$
KF	$6.1 \times 10^{-2}$	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$	$1.1 \times 10^{-6}$
NaF	$5.5 \times 10^{-2}$	$3.4 \times 10^{-6}$	$1.2 \times 10^{-5}$	$8.0 \times 10^{-7}$
BLANK	—	$2.0 \times 10^{-6}$	$1.2 \times 10^{-5}$	$1.7 \times 10^{-7}$

Note: Underlined figures are 1 molal solutions; all others became saturated solutions at less than a 1 molal concentration.

best presently known nonaqueous systems are approximately  $10^{-2}$  to  $5 \times 10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$ . Thus the development of improved electrolytes is the key to new high energy batteries.

At very low concentrations the behavior of many different ionizable solutes is quite uniform in a given solvent, the conductivity level thus being determined by the properties of the solvent itself. As concentration increases, major differences in conductivity appear, apparently dependent mainly upon limits of solubility. Solubility appears to increase with increasing ion (or solvated ion) size, but other factors may be important. Much remains to be done in the elucidation of this aspect of the problem.

### Symbols

E°	= Standard electrode potential in volts
WH/LB	= Watt-hours per pound
BN	= Benzonitrile
DMF	= Dimethylformamide
FA	= Formamide
NMF	= N-Methylformamide
PC	= Propylene Carbonate
TBAI	= Tetra Butyl Ammonium Iodide
$\epsilon$	= Dielectric Constant
$\eta$	= Viscosity in Centipoises
$\Lambda^\circ$	= Equivalent Conductance at infinite dilution
$\Lambda_{\text{max}}$	= Equivalent Conductance at $L_{\text{max}}$ concentration
$L_{\text{max}}$	= Maximum Specific Conductance
$\bar{\Lambda}^\circ$	= Average $\Lambda^\circ$
$C_{L_{\text{max}}}$	= Molal concentration at $L_{\text{max}}$

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Los Angeles 45, California

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Attention: Frank J. Mollura

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Attention: Marshall D. Aiken (AMCRD-DE-MO-P)

Mr. Donald B. Hoatson  
Army Reactors DRD  
U. S. Atomic Energy Commission  
Washington 25, D. C.

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Naval Ammunitions Depot  
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Attention: E. Bruess/H. Shultz

U. S. Naval Research Laboratory  
Washington, D. C. 20390  
Attention: Code 6160

Captain William H. Ritchie  
Space Systems Division  
Attention: SSZAE-11  
Air Force Unit Post Office  
Los Angeles 45, California

Office of the Deputy Commander  
AFSC, Aerospace Systems  
U. S. Air Force  
Los Angeles 45, California  
Attention: W. J. Bennison

Aerospace Corporation  
P. O. Box 95085  
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Attention: LCOL George H. Ogburn, Jr.

Power Information Center  
University of Pennsylvania  
Moore School Building  
200 South 33rd Street  
Philadelphia 4, Pennsylvania

Office, DDR&E; USW and BSS  
The Pentagon  
Washington 25, D. C.  
Attention: Mr. G. B. Wareham

Institute of Defense Analysis  
1666 Connecticut Avenue, N.W.  
Washington 9, D. C.  
Attention: Dr. J. Szego/Mr. R. Hamilton

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Missile & Space Division  
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Philadelphia 1, Pennsylvania  
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Minneapolis 14, Minnesota  
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Central Research Laboratory  
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Philadelphia 24, Pennsylvania  
Attention: Dr. H. L. Tfluger

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Battery Products Section  
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Gainsville, Florida

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Columbus 1, Ohio  
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Anderson, Indiana  
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466 South Center Street  
Orange, New Jersey

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Metuchen, New Jersey  
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Santa Barbara, California  
Attention: Dr. J. S. Smatko

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Milwaukee, Wisconsin 53201  
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Attention: Mr. T. M. Hahn

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Sunnyvale, California  
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52-30/Larry E. Nelson,  
Dept. 65-82

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Attention: Dr. R. Selim

Marquardt Corporation  
16555 Saticoy Street  
Van Nuys, California  
Attention: Dr. H. G. Krull

American University  
Massachusetts & Nebraska Avenues, N.W.  
Washington, D. C. 20016  
Attention: Dr. R. T. Foley, Chem. Dept.

Midwest Research Institute  
425 Volker Boulevard  
Kansas City 10, Missouri  
Attention: Dr. B. W. Beadle

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Everett 49, Massachusetts  
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University of Pennsylvania  
Electrochemistry Laboratory  
Philadelphia 4, Pennsylvania  
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Somerville, New Jersey  
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Rocketdyne Division  
6633 Canoga Avenue  
Canoga Park, California

Southwest Research Institute  
8500 Culebra Road  
San Antonio 6, Texas  
Attention: Dr. Jan Al

Power Sources Division  
Whittaker Corporation  
9601 Canoga Avenue  
Chatsworth, California 91311  
Attention: Dr. M. Shaw

Electro Optical Systems  
300 North Halstead  
Pasadena, California  
Attention: E. Findl

Thomas A. Edison Research Laboratory  
McGraw Edison Company  
Watchung Avenue  
West Orange, New Jersey  
Attention: Dr. P. F. Grieger

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New York 13, New York  
Attention: Dr. P. L. Howard

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R&D Directorate  
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Cleveland, Ohio 44101

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Lake Success Park  
Community Drive  
Great Neck, New York 11021  
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123 N. Mathilda Avenue  
Sunnyvale, California  
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Indianapolis 6, Indiana  
Attention: A. S. Doty

North American Aviation  
12214 Lake Wood Boulevard  
Downey, California  
Attention: Burton M. Otzinger

Dr. John Owen  
P. O. Box 87  
Bloomfield, New Jersey

Radiation Application, Inc.  
36-40 37th Street  
Long Island City, New York  
Attention: Monroe F. Pofcher

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Heightstown, New Jersey  
Attention: Seymour Winkler

Radio Corporation of America  
P. O. Box 800  
Princeton, New Jersey  
Attention: Paul Wiener

Radio Corporation of America  
Sommerville, New Jersey  
Attention: Semiconductor & Material  
Division

Sonotone Corporation  
Saw Mill River Road  
Elmsford, New York  
Attention: A. Mundel

Space Technology Laboratories  
2400 East ElSegundo Boulevard  
ElSegundo, California  
Attention: A. Krausz

Power Sources Division  
Whittaker Corporation  
Denver, Colorado  
Attention: Borch Winder